

Appendix F

Data Validation Reports



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC18120/JC18120A
Sample Dates: April 12, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Methods 3060A/7196A
Trivalent Chromium Analysis, EPA Methods 6010/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: May 20, 2016

This data validation (DV) report presents the data review and result qualifications for six (6) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on April 12, 2016, for sample delivery group (SDG) JC18120, as well as JC18120A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC18120A and JC18120 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium (Cr+6), as well as trivalent chromium (Cr+3) in the 6 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC18120-2A through JC18120-6A (inclusive)

No other sample results in SDG JC18120A and JC18120 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below. No hexavalent or trivalent chromium results for the 6 soil samples of SDG JC18120 were qualified following the DV review, because all QC results were within method QC limits.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever were more stringent, except the nickel results in three samples (JC18120-2A, -5A, and -6A), while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Table 4 of this DV report.

Sample Receipt

The six (6) post-excavation soil samples collected April 12, 2016, were received intact and preserved appropriately the same day, April 12, at the Accutest laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 4.9 degrees Celsius.. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC18120A and JC18120

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-EPG-CC01	JC18120-1A	4/12/16	Soil	Metals, Cr+3
PPG174-EPG-B01	JC18120-2A	4/12/16	Soil	Metals, Cr+3
PPG174-EPG-SW01	JC18120-3A	4/12/16	Soil	Metals, Cr+3
PPG174-EPG-SW02	JC18120-4A	4/12/16	Soil	Metals, Cr+3
PPG174-EPG-SW03	JC18120-5A	4/12/16	Soil	Metals, Cr+3
PPG174-EPG-SW04	JC18120-6A	4/12/16	Soil	Metals, Cr+3
PPG174-EPG-CC01	JC18120-1	4/12/16	Soil	Cr+6
PPG174-EPG-B01	JC18120-2	4/12/16	Soil	Cr+6
PPG174-EPG-SW01	JC18120-3	4/12/16	Soil	Cr+6
PPG174-EPG-SW02	JC18120-4	4/12/16	Soil	Cr+6
PPG174-EPG-SW03	JC18120-5	4/12/16	Soil	Cr+6
PPG174-EPG-SW04	JC18120-6	4/12/16	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. Cr+3 – Trivalent chromium calculated as: Cr+3 = (chromium) – (Cr+6)				

The data package presenting the metals and Cr+3 data is numbered JC18120A, while the data package for the hexavalent chromium analyses is numbered JC18120.

Data Review

Data, as presented in the analytical data packages SDG JC18120A and JC18120, was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.

- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC18120A

The data validation of the metals analytical data in SDG JC18120A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 6 post-excavation soil samples were analyzed for the 5 target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 6 samples of SDG JC18120A, the nickel results in three samples (JC18120-2A, -5A, and -6A) exhibited a concentration above the IGWSSL of 48 milligrams per kilogram (mg/kg) for nickel.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP93102 indicating possible matrix interference and/or sample non-homogeneity. The case narrative identified the serial dilution result being outside QC limits for antimony in QC Batch 93102 and antimony,

chromium and thallium in QC Batch MP93116; however, the percent difference (%D) results were acceptable due to low initial sample concentrations (< 50 times Instrument Detection Limit [IDL]). The case narrative also stated that the detection limit for thallium was elevated in samples JC18120-2A, -5A, and -6A due to dilution required for the presence of a high interfering element. All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

The results for trivalent chromium (Cr+3) were also reported for the samples of this SDG. The Cr+3 results were calculated as follows: $Cr+3 = (\text{Chromium}) - (Cr+6)$

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

One exception consisted of the 0% recovery of thallium in CRID1 at 16:33 in analytical sequence MA39166 associated with only QC samples. However, the soil sample results were not affected because the reporting limits for thallium are above the respective affected range where results may be subject to qualification. The affected range is approximately 0 – 0.44 mg/kg for thallium where the corresponding reporting limits of approximately 1 mg/kg are above the affected range of thallium and sample results are, hence, not subject to qualification.

The exceptions also consisted of the 175%, 145%, and 210% recoveries of thallium in CRID1 at 11:51, CRID2 at 18:26, and CRID3 at 1:05 in analytical sequence MA39173 associated with only Sample JC18120-1A and the associated QC samples which were run after CRID3. However, these elevated contract required detection limit (CRDL) standard recoveries did not affect the thallium results because thallium was not detected in the associated method and instrument blanks, and thallium was not detected in any of the soil samples of this SDG. The non-detect thallium results are not subject to qualification, as there is no positive bias in a non-detected result. Thus, no sample results required qualification for calibration issues.

Consequently, no soil sample or QC blank results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), except that thallium was detected in CCB10 at 21:07 at a concentration of 2.5 micrograms per liter (µg/L) and CCB14 at 1:05 at a concentration of 2.2 µg/L in analytical sequence MA39173. However, the detection of thallium in these CCBs did not affect the soil sample results, since these two CCBs were not directly associated with Sample JC18120-1A or the QC samples in this analytical sequence and thallium was not detected in the samples of this SDG. Hence, no soil sample results warranted qualification for any associated QC blank contamination in SDG JC18120A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35% Relative Percent Difference [RPD])

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for a non-client QC batch sample, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits, including those of QC Batch GP93116 associated with Sample JC18120-1A.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93102 Ω	JC18063-2	Antimony	48.2 %	48.5 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ω – The samples associated with QC Batch MP93102 consist of JC1812-2A through -6A (inclusive).						

The antimony results in these five affected soil samples are flagged with “NJ-” due to a potential low bias. The metals concentrations in the non-client QC sample appear to be similar to those typically observed in PPG samples and, therefore, qualification of the associated antimony results was judged appropriate in this case. The qualified antimony results are presented below in the summary table, Table 4.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on two pairs of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 0.4 – 1.6%RPD for five soil samples and 2.9 – 3.9 %RPD for the batch QC sample associated with the remaining soil sample JC18120-1A with no results requiring qualification. The duplicate analyses demonstrated excellent analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 93.5% - 103.0% for the soil sample metals analysis covering both QC batches.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative stated that the RPD serial dilution results for antimony in QC Batch MP93102, and antimony, chromium, and thallium were outside control limits in QC Batch MP93116; however, the percent difference (%D) results were acceptable due to a low initial sample concentrations (< 50 times IDL). The remaining serial dilution results associated with the soil samples ranged from 0

– 8.7%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

The case narrative did identify that there were three samples (JC18120-2A, -5A, and -6A) with thallium reporting limits that were elevated due to the presence of a “high interfering element.” Review of the data indicated that there were a total of three soil samples with thallium that had been diluted for analysis of metals at a dilution factor of either 2× or 3×. The reporting limits for the target analytes determined for the ICP metals analysis employing the various assigned laboratory instruments all were below the respective site remediation standards; the exception being the thallium result that was diluted by a factor of 3× in Sample JC18120-5A, thereby raising the reporting limit for the thallium result above the respective IGWSSL criterion, as detailed below in Table 3. The samples were diluted likely due to the presence of an interfering element.

Table 3. Sample Reporting Limits Affected by Sample Dilution

Sample ID	Lab ID	Analyte	Reporting Limit (mg/kg)	Dilution Factor	Adjusted Result (mg/kg)	Remediation Standard (mg/kg)
PPG174-EPG-B01	JC18120-2A	Thallium	< 1.15	2	< 2.3	3
PPG174-EPG-SW03	JC18120-5A	Thallium	< 1.133	3	< 3.4	3
PPG174-EPG-SW04	JC18120-6A	Thallium	< 1.15	2	< 2.3	3
Units – mg/kg (milligrams per kilogram)						
< - The analyte was analyzed for but was not detected above the stated reporting limit.						

The interpretation of the non-detect thallium results in these three samples, including Sample JC18120-5A whose reporting limit was above the IGWSSL of 3 mg/kg, were not compromised by the applied 2× or 3× dilution, because the nickel concentrations were above the IGWSSL of 48 mg/kg in each of these three soil samples. Consequently, these soil samples would be, thus, potentially subject to some sort of response action or further evaluation, not related to the thallium results.

Summary of Qualified Metals Results

The post-excavation soil sample analytical results for the samples of SDG JC18120A were found to be compliant with the analytical methods for the analysis of metals in the 6 soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP93102 associated with five of the six soil samples: JC18120-2A through JC18120-6A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 4.

Table 4. Summary of Qualified Sample Metals Results in SDG JC18120A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-EPG-B01	JC18120-2A	Antimony	< 2.3	NJ-
PPG174-EPG-SW01	JC18120-3A	Antimony	< 2.3	NJ-

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-EPG-SW02	JC18120-4A	Antimony	< 2.2	NJ-
PPG174-EPG-SW03	JC18120-5A	Antimony	< 2.3	NJ-
PPG174-EPG-SW04	JC18120-6A	Antimony	< 2.3	NJ-

Key:

< –The analyte was analyzed for but was not detected above the stated reporting limit.
 NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.
 J – The result is an estimated value.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC18120

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the six post-excavation soil samples.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- √ Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Data qualifiers

Hexavalent chromium was detected in 5 of the 6 soil samples analyzed in SDG JC18120, with all sample Cr+6 results less than 10 mg/kg, all values below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time, method blanks, as well as matrix spike recoveries. The RPD(s) for the duplicate analysis was outside [laboratory] control limits for Cr+6, due possibly to sample non-homogeneity. No other QC requirements were exceeded.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99984 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 94.2% to 94.5% for the QC batch associated with the analysis of 6 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The matrix spike and matrix spike duplicate (MS/MSD) recoveries for hexavalent chromium were all within QC limits of 75 - 125% for PPG sample JC18120-3 associated with the soil samples, such that no soil sample results were qualified for matrix spike recoveries, thereby indicating acceptable analytical accuracy in the ability to recover Cr+6 in the associated sample matrices, as demonstrated in Table 5.

Table 5. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC18120

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP96894 ¥	JC18120-3	Cr ⁺⁶ , soluble	98.3 %	----	----
GP96894 ¥	JC18120-3	Cr ⁺⁶ , insoluble	106.1 %	----	----
GP96894 ¥	JC18120-3	Cr ⁺⁶ , post-digestion spike	85 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium ¥ – The samples associated with QC Batch GP96894 consist of JC18120-1 through -6 (inclusive).					

Because of the acceptable MS recoveries, no Cr+6 results required qualification in the soil sample analysis.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from sample JC18120-3 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-EPG-SW01) was 29.6%RPD, a value above the 20%RPD laboratory QC limit, but within the 35%RPD DV advisory QC limit for technical review of soil sample data (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (2.6%RPD) and pH (0.8%RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 89.8% and 95.9% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)
 C = Wet weight of sample (kg)
 D = % Solids/100
 E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-EPG-SW01 (JC18120-3) was listed as 9.3 mg/kg on the reporting form and 0.1988 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.1988 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00259 \text{ Kg} \times 82.3/100} = \frac{0.01988}{0.0021316} = 9.3265 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 9.3 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 9.3 mg/kg for Sample PPG174-EPG-SW01 was correctly reported. This was the highest detected Cr+6 concentration of the 5 detected results for the 6 soil samples of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Five of the six soil samples were observed to fall above the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of an “oxidizing” soil environment. The Cr+6 sample results in a “reducing” soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions, while Cr+6 may increase under “oxidizing” conditions, provided there is a significant concentration of chromium available. The sample Cr+6 concentrations detected in five of the six samples are all below 10 mg/kg, with total chromium concentrations for the samples with detected Cr+6 all less than 140 mg/kg. The non-detect result in JC18120-5 fell below the Eh-pH phase diagram line indicating a “reducing” soil character with a total chromium result of 248 mg/kg but is not expected to increase to levels approaching the SCC of 20 mg/kg, because of the reducing soil matrix indicated by the Eh-pH results. It has been observed that PPG soil samples with total chromium concentrations less than 600 mg/kg in a

reducing soil do not exhibit Cr+6 concentrations that would increase to any significant degree that approach the SCC of 20 mg/kg.

There were five soil samples that were observed to fall above the Eh-pH phase diagram line in JC18120. The chromium to Cr+6 ratios (Cr:Cr+6) in these five samples representing “oxidizing” conditions ranged from approximately 14 – 38, similar to ratios observed in many other samples in the PPG project, irrespective of Redox state.

Summary for Hexavalent Chromium Analysis – SDG JC18120

Since the QC requirements were met in the soil samples, no Cr+6 results were subject to qualification.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

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US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC18120/JC18120A

1. Were the appropriate sample preservation requirements met?..... Yes No
2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Samples JC18120-2A and -6A were diluted 2× for thallium analysis, while JB18120-5A was diluted 3×, due to the presence of a high interfering element.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

The reporting limit for thallium in Sample JC18120-5A (< 3.4 mg/kg) exceeded the IGWSSL of 3 mg/kg.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The nickel results in Samples JC18120-2A, -5A, and -6A exceeded the IGWSSL of 48 mg/kg.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.
Not applicable
10. Were rejected data used?..... Yes **No**
If "yes", please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report Tables 2 and 5 for QC details. Qualified sample results are presented in Table 4 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC18222/JC18222A
Sample Dates: April 12, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Trivalent Chromium Analysis, EPA Methods 6010/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: May 20, 2016

This data validation (DV) report presents the data review and result qualifications for five (5) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on April 12, 2016, for sample delivery group (SDG) JC18222, as well as JC18222A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC18222A and JC18222 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 5 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC18222-1A through JC18222-5A (inclusive)
- Hexavalent chromium ("NJ-") in Samples JC18222-1 through JC18222-5 (inclusive)
- Hexavalent chromium ("NJ-") in reanalysis samples JC18222-1R through JC18222-5R (inclusive)
- Total organic carbon ("NJ+") in Samples JC18222-1RT

No other sample results in SDG JC18222A and JC18222 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except the nickel results in each of the five samples (JC18222-1A, -2A, -3A, -4A, and -5A), while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Tables 4 and 7 of this DV report.

Sample Receipt

The five (5) post-excavation soil samples collected April 12, 2016, were received intact and appropriately preserved on April 13 at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 2.8 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC18222A and JC18222

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-WPG-B01	JC18222-1A	4/12/16	Soil	Metals, Cr+3
PPG174-WPG-SW01	JC18222-2A	4/12/16	Soil	Metals, Cr+3
PPG174-WPG-SW02	JC18222-3A	4/12/16	Soil	Metals, Cr+3
PPG174-WPG-SW03	JC18222-4A	4/12/16	Soil	Metals, Cr+3
PPG174-WPG-SW04	JC18222-5A	4/12/16	Soil	Metals, Cr+3
PPG174-WPG-B01	JC18222-1	4/12/16	Soil	Cr+6
PPG174-WPG-B01	JC18222-1RT	4/12/16	Soil	TOC, SS, Fe2+
PPG174-WPG-SW01	JC18222-2	4/12/16	Soil	Cr+6
PPG174-WPG-SW02	JC18222-3	4/12/16	Soil	Cr+6
PPG174-WPG-SW03	JC18222-4	4/12/16	Soil	Cr+6
PPG174-WPG-SW04	JC18222-5	4/12/16	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. Cr+3 – Trivalent chromium calculated as: Cr+3 = (chromium) – (Cr+6). TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting the metals and trivalent chromium data is numbered JC18222A, while the data package for the hexavalent chromium analyses is numbered JC18222. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC18222 together with the supplemental total organic carbon (TOC), sulfide screen and ferrous iron. The samples data were validated for the five target metals (antimony, chromium, nickel, thallium, and vanadium), as were the hexavalent chromium data, and supplemental TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC18222A and JC18222, was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002),
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC18222A

The data validation of the metals analytical data in SDG JC18222A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 5 post-excavation soil samples were analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 5 samples of SDG JC18222A, the nickel results in each of the five samples (JC18222-1A, -2A, -3A, -4A, and -5A) exhibited a concentration above the IGWSSL of 48 milligrams per kilogram (mg/kg) for nickel.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP93102 indicating possible matrix interference and/or sample non-homogeneity. The case narrative identified the serial dilution result being outside QC limits for antimony in QC Batch 93102; however, the percent difference (%D) result was acceptable due to the low initial sample concentration (< 50 times Instrument Detection Limit [IDL]). The case narrative also stated that the detection limit for thallium was elevated in samples JC18222-1A, -2A, and -3A due to dilution required for the presence of a high interfering element. All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and "low calibration check standard" ("CRI" standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

One exception consisted of the 0% recovery of thallium in CRID1 at 16:33 in analytical sequence MA39166 associated with only QC samples. However, the soil sample results were not affected because the reporting limits for thallium are above the respective affected range where results may be subject to qualification. The affected ranges are approximately 0 – 0.44 mg/kg for thallium where the corresponding reporting limits of approximately 1 mg/kg are above the affected range of thallium and sample results are, hence, not subject to qualification.

Consequently, no soil sample or field blank results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC18222A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35% Relative Percent Difference [RPD])

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for a non-client QC batch sample, JC18063-2, as identified in Table 2

below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (USEPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93102 Ω	JC18063-2	Antimony	48.2 %	48.5 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ω – The samples associated with QC Batch MP93102 consist of JC18222-1A through -5A (inclusive).						

The antimony results in the five affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 4.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 0.4 – 1.6%RPD for QC sample associated with the soil sample analysis with no results requiring qualification. The duplicate analyses demonstrated excellent analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 97.5% - 105.0% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative stated that the RPD serial dilution result for antimony in QC Batch MP93102 was outside control limits, however, the percent difference (%D) result was acceptable due to a low initial sample concentration (< 50 times IDL). The remaining serial dilution results associated with the soil samples ranged 0 – 8.7%D, values below the QC limit of 10%D criterion for data validation qualification (USEPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

The case narrative did identify that there were three samples (JC18222-1A, -2A, and -3A) with thallium reporting limits that were elevated due to the presence of a “high interfering element.” Review of the data indicated that there were a total of three soil samples with thallium that had been diluted for analysis of metals at a dilution factor of either 2× or 3×. The reporting limits for the

target analytes determined for the ICP metals analysis employing the various assigned laboratory instruments all were below the respective site remediation standards; the exceptions being the thallium results that were diluted by a factor of 3× in Samples JC18222-2A and -3A, thereby raising the reporting limit for the thallium result above the respective IGWSSL criterion, as detailed below in Table 3.

Table 3. Sample Reporting Limits Affected by Sample Dilution

Sample ID	Lab ID	Analyte	Reporting Limit (mg/kg)	Dilution Factor	Adjusted Result (mg/kg)	Remediation Standard (mg/kg)
PPG174-WPG-B01	JC18222-1A	Thallium	< 1.05	2	< 2.1	3
PPG174-WPG-SW01	JC18222-2A	Thallium	< 1.1	3	< 3.3	3
PPG174-WPG-SW02	JC18222-3A	Thallium	< 1.13	3	< 3.4	3

Units – mg/kg (milligrams per kilogram)
 < - The analyte was analyzed for but was not detected above the stated reporting limit.

The interpretation of the non-detect thallium results in these three samples, including Samples JC18222-2A and -3A whose reporting limits are above the IGWSSL of 3 mg/kg, were not compromised by the applied 2× or 3× dilution, because the nickel concentrations were above the IGWSSL of 48 mg/kg in each of these three soil samples. Consequently, these soil samples would be, thus, potentially subject to some sort of response action or further evaluation.

Summary of Qualified Metals Results

The post-excavation soil sample analytical results for the samples of SDG JC18222A were found to be compliant with the analytical methods for the analysis of metals in the 5 post-excavation soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP93102 associated with the five soil samples: JC18222-1A through JC18222-5A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias in the ability to recover antimony from the soil matrix, as summarized below in Table 4.

Table 4. Summary of Qualified Sample Metals Results in SDG JC18222A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-WPG-B01	JC18222-1A	Antimony	< 2.1	NJ-
PPG174-WPG-SW01	JC18222-2A	Antimony	< 2.2	NJ-
PPG174-WPG-SW02	JC18222-3A	Antimony	< 2.2	NJ-
PPG174-WPG-SW03	JC18222-4A	Antimony	< 2.4	NJ-
PPG174-WPG-SW04	JC18222-5A	Antimony	< 2.1	NJ-

Key:
 mg/kg - milligrams per kilogram
 < –The analyte was analyzed for but was not detected above the stated reporting limit.
 NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC18222

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the five post-excavation soil samples.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was detected in each of the five post-excavation soil samples analyzed in SDG JC18222, with all sample Cr+6 results less than or equal to 3.0 mg/kg, all values below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery in QC Batch GP96925 was outside control limits, as was the soluble MS recovery in reanalysis batch GP96943. The RPD values for the duplicate analyses in QC batches GP96925 and the re-analysis batch GP96943. The matrix spike recovery for the TOC analysis of non-client sample JC17896-5 was also outside control limits. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99969 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 90.9% to 91.1% for the QC batch associated with the analysis of 5 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP96925 associated with the 5 soil samples of this SDG, as presented below in Table 5. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP96925 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. All remaining MS recoveries were within QC limits.

Table 5. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC18222

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP96925 ¥	JC18222-1	Cr ⁺⁶ , soluble	58.0 %	NJ-	Low
GP96925 ¥	JC18222-1	Cr ⁺⁶ , insoluble	95.9 %	----	----
GP96925 ¥	JC18222-1	Cr ⁺⁶ , post-digestion spike	93 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. ¥ – The samples associated with QC Batch GP96925 consist of JC18222-1 through -5 (inclusive).					

The Cr+6 results qualified for low soluble matrix spike recovery are flagged with “NJ-”, as tabulated below in Table 7, together with the qualified results from the re-analysis of this QC batch. The Cr+6 results in the 5 soil samples in are qualified as estimated values because the soluble MS recovery was within the 50-75% QC range where DV guidelines recommend qualification of associated samples results to be flagged with “J” due to a potential low bias (NJDEP, 2009).

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from sample JC18222-1 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-WPG-B01) was 26.1%RPD, a value above the 20%RPD laboratory QC limit, but within the 35%RPD DV advisory QC limit for technical review of soil sample data (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (4.0%RPD) and pH (0.4%RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 88.7% and 89.0% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)

B = Final digested volume (L)
C = Wet weight of sample (kg)
D = % Solids/100
E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-WPG_B01 (JC18222-1) was listed as 3.0 mg/kg on the reporting form and 0.0697 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0697 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00254 \text{ Kg} \times 90.0/100} = \frac{0.00697}{0.0022860} = 3.0489 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 3.0 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 3.0 mg/kg for Sample PPG174-WPG_B01 was correctly reported. This was the highest detected Cr+6 concentration of the five detected results for the 5 soil samples of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

All five soil samples were observed to clearly fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentrations are also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total chromium concentrations are all less than 240 mg/kg, thereby making it unlikely that Cr+6 concentrations would increase to any significant degree. Following review of many soil samples from various PPG sites, it has been observed that PPG soil samples with total chromium concentrations less than 600 mg/kg do not exhibit Cr+6 concentrations that would increase to any significant degree that approach the SCC of 20 mg/kg.

Hence, based on the sample total chromium and Cr+6 concentrations, it is unlikely that any of the affected samples including those in the “reducing” zone would approach the SCC for Cr+6 of 20 mg/kg due to limitation created by the relatively low total chromium concentrations available for potential oxidation.

Cr+6 Re-analyses in SDG JC18222

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the re-analysis batch consisting of 5 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC18222-1R through -5R in QC Batch GP96943, including the calibrations (r = 0.99984, 96.9 – 97.4% CCV Recoveries), QC blanks,

duplicate analysis ($\leq 2 \times$ Contract Required Quantitation Limit), and blank spike analysis (91.5% – 93.0%). The soluble MS recovery was slightly higher in the reanalysis, but still below QC limits, while the post spike was also slightly lower, but just meeting QC limits, as detailed below. There was not a good agreement between the samples and the 1:5 dilution.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, all but the post-digestion spike recovery in QC Sample JC18222-1R were all slightly higher compared to the initial analyses, as observed below in Table 6. The insoluble MS recovery in JC7615-1R was still well within the 75-125% QC limits.

Table 6. Hexavalent Chromium Re-analysis MS Recovery Results – JC18222

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP96943 Ж	JC18222-1R	Cr ⁺⁶ , soluble	61.5 %	NJ-	Low
GP96943 Ж	JC18222-1R	Cr ⁺⁶ , insoluble	96.7 %	----	----
GP96943 Ж	JC18222-1R	Cr ⁺⁶ , post-digestion spike	84.6 %	----	----
GP96943 Ж	JC18222-1R	Cr ⁺⁶ , pH-adjusted post spike	91.2 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
 MS – Matrix spike
 Cr⁺⁶ – Hexavalent chromium
 NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.
 Ж – The samples associated with QC Batch GP96943 consist of JC18222-1R through -5R (inclusive).

Since the soluble MS recovery in QC Batch GP96943 was still below the QC limits (75-125%), the Cr⁺⁶ results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” for a potential low bias in the ability to recover Cr⁺⁶ in this QC batch. Data validation guidelines for Cr+6 analysis recommend qualifying results as estimated values when associated with MS recoveries ranging 50-75% (NJDEP, 2009). The qualified Cr+6 results of the reanalysis are presented below in Table 7 together with the results of the initial Cr+6 results.

The post-digestion spike recovery value rounds to 85%, just meeting the QC limit. The note on the Matrix Spike Recovery Summary sheet stated “Low pH adjusted spike (91%).” However, this statement is incorrect, since 91% falls within the QC limit range of 85-115% for post-digestion spikes.

Duplicate Sample Analysis

The duplicate analyses were performed on one set of duplicate soil sample aliquots. The differences between the duplicate soil sample aliquot concentrations for Cr+6 in the sample aliquots was listed as 200%RPD. Although this RPD value exceeded 35%, the analytical precision results were acceptable because of the low sample concentrations where the difference (0.654 mg/kg) between the raw concentration values was less than twice the reporting limit (2×0.44 mg/kg). Hence, the Cr+6 results in the associated samples were not qualified for the duplicate analysis result and analytical precision is considered acceptable in the re-analysis.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC18222-1RT (PPG174-WPG_B01), a QC samples which was analyzed twice with detected concentrations of 3.0 and 1.0 mg/kg for the analyses, values well below the SCC of 20 mg/kg. The

ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr⁺⁶ recoveries. The associated QC results were all within the respective QC limits. Professional judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. The matrix spike recovery of 133% in the batch QC sample in the total organic carbon (TOC) analysis was outside the listed QC range of 39.6 – 124%. Hence, the TOC result is qualified as an estimated value and flagged with “NJ+” in Table 7, below, due to a potential positive bias. In accordance with the method, these analyses were performed on the sample experiencing the low spike recoveries. A concentration of TOC (6,930 mg/kg) and ferrous iron (Fe⁺²) with a result of 2.1 % were detected in the QC sample in JC18222-1RT, thereby indicating the likely presence of a reducing soil matrix in the soil sample, as suggested by the presence of this soil sample below the Eh-pH phase line, as are the other four soil samples of this SDG.

The “reducing” conditions in the soil matrix appear supported by the detected TOC concentration and the detected Fe⁺² data in support of the results of the Eh-pH analyses.

Summary for Hexavalent Chromium Analysis – SDGs JC18222

The qualified soil sample results from the initial Cr+6 analysis in SDG JC18222 are presented below in Table 7 alongside those qualified results obtained from the reanalysis of the samples. Both sets of analytical Cr+6 results for samples JC18222-1 through -5 and their reanalysis are still both qualified as estimated values (NJ-) due to a potential low bias, although the soluble MS recoveries of the second analysis exhibited slightly higher recoveries in the re-analyses that were performed within the 30-day holding time. The Cr+6 concentrations determined during the re-analysis of samples in SDG JC18222 differ slightly from those of the initial analysis, but all are still well below the SCC of 20 mg/kg.

Table 7. Comparison of Qualified Cr+6 Results in JC18222 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC18222 Result (mg/kg)	DV Qualifier	JC18222-R Results (mg/kg)	DV Qualifier
PPG174-WPG-B01	JC18222-1	Cr+6	3.0	NJ-	1.0	NJ-
PPG174-WPG-B01	JC18222-1RT	TOC	----	----	6,930	NJ+
PPG174-WPG-SW01	JC18222-2	Cr+6	1.1	NJ-	0.53	NJ-
PPG174-WPG-SW02	JC18222-3	Cr+6	0.84	NJ-	0.78	NJ-
PPG174-WPG-SW03	JC18222-4	Cr+6	1.0	NJ-	1.2	NJ-
PPG174-WPG-SW04	JC18222-5	Cr+6	1.7	NJ-	2.2	NJ-
mg/kg - milligrams per kilogram NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. NJ+ – The matrix spike sample recovery in the associated QC sample is above QC limits; the result is estimated and may be biased high.						

Professional judgement was applied in qualifying the Cr+6 results in both analyses as estimated values (NJ-) due to a potential low bias, as suggested by the MS results tabulated above in Table 5 and Table 6, an approach consistent with DV guidelines (NJDEP, 2009) which recommend qualifying associated sample results when MS recoveries range 50-75%. The samples in SDG JC18222 exhibited low concentrations for Cr+6 in the analysis associated with the low soluble MS recoveries. These samples exhibited corresponding total chromium results less than 240 mg/kg, making it unlikely that sample Cr+6 concentrations would approach the SCC of 20 mg/kg for these samples experiencing “reducing” soil conditions, which do not favor oxidation to Cr+6. Additionally,

the insoluble MS recoveries were within QC limits for both the initial and re-analyses and may be a better representation of the ability of the analysis to recover Cr+6 from the soil matrix than the soluble MS recovery result.

Although the samples were re-analyzed within the 30-day holding time, the Cr+6 concentrations differed slightly upon reanalysis. However, all Cr+6 sample results exhibited a Cr+6 values considerably below the SCC of 20 mg/kg, consistent with the redox state of the sample's soil environment.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
NJ+	The matrix spike sample recovery in the associated QC sample is above QC limits; the result is estimated and may be biased high

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

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ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC18222/JC18222A

1. Were the appropriate sample preservation requirements met?..... Yes No

2. Were appropriate sample holding times (for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Sample JC18222-1A was diluted 2x for thallium analysis, while JB18222-2A and -3A were diluted 3x, due to the presence of a high interfering element.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

The reporting limit for thallium in Samples JC18222-2A (< 3.3 mg/kg) and -3A (< 3.4 mg/kg) exceeded the IGWSSL of 3 mg/kg.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The nickel results in Samples JC18222-1A, -2A, -3A, -4A, and -5A exceeded the IGWSSL of 48 mg/kg.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.
Not applicable
10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2, 5, and 6 for QC details. Qualified sample results are presented in Tables 4 and 7 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC18361/JC18361A
Sample Dates: April 13 – 14, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Trivalent chromium, Method 6010C/7196A M
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11

Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: May 20, 2016

This data validation (DV) report presents the data review and result qualifications for seven (7) post-excavation soil samples and one (1) field blank (FB) collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, from April 13 to 14, 2016, for sample delivery group (SDG) JC18361, as well as JC18361A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC18361A and JC18361 were found to be compliant with the analytical methods employed for the analysis of metals, hexavalent chromium (Cr+6), and trivalent chromium (Cr+3) in the 7 collected post-excavation soil samples and one field blank.

Following the detailed DV review, the following sample results were qualified:

- Antimony (“NJ-”) in Samples JC18361-2A through JC18361-8A (inclusive)
- Chromium, nickel and vanadium (“EJ”) in Samples JC18361-2A through JC18361-8A (inclusive)
- Trivalent chromium (“J”) in Samples JC18361-2A through JC18361-8A (inclusive)
- Hexavalent chromium (“NJ-”) in Samples JC18361-2 through JC18361-8 (inclusive)
- Hexavalent chromium (“NJ-”) in reanalysis samples JC18361-2R through JC18361-8R (inclusive)

No other sample results in SDG JC18361A and JC18361 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in tables and text below. No field blank results were qualified following the DV review, because the QC results were within method QC limits.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever

was more stringent, except the nickel in JC18361-6A and vanadium in JC18361-5A, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Tables 5 and 8 of this DV report.

Sample Receipt

The seven (7) soil samples and one field blank collected April 13 and 14, 2016, were received intact and appropriately preserved April 14, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 4.5 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC18361A and JC18361

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174 FB01	JC18361-1A	4/13/2016	Aqueous	Metals
PPG174 BERM B01	JC18361-2A	4/14/2016	Soil	Metals, Cr+3
PPG174 BERM B02	JC18361-3A	4/14/2016	Soil	Metals, Cr+3
PPG174 BERM SW01	JC18361-4A	4/14/2016	Soil	Metals, Cr+3
PPG174 BERM SW02	JC18361-5A	4/14/2016	Soil	Metals, Cr+3
PPG174 BERM SW03	JC18361-6A	4/14/2016	Soil	Metals, Cr+3
PPG174 BERM SW04	JC18361-7A	4/14/2016	Soil	Metals, Cr+3
PPG174 BERM SW05	JC18361-8A	4/14/2016	Soil	Metals, Cr+3
PPG174 FB01	JC18361-1	4/13/2016	Aqueous	Cr+6
PPG174 BERM B01	JC18361-2	4/14/2016	Soil	Cr+6
PPG174 BERM B01	JC18361-2RT	4/14/2016	Soil	TOC, SS, Fe2+
PPG174 BERM B02	JC18361-3	4/14/2016	Soil	Cr+6
PPG174 BERM SW01	JC18361-4	4/14/2016	Soil	Cr+6
PPG174 BERM SW02	JC18361-5	4/14/2016	Soil	Cr+6
PPG174 BERM SW03	JC18361-6	4/14/2016	Soil	Cr+6
PPG174 BERM SW04	JC18361-7	4/14/2016	Soil	Cr+6
PPG174 BERM SW05	JC18361-8	4/14/2016	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. Cr+3 – Trivalent chromium calculated as (Chromium – Cr+6) from Methods 6010C and 7196A. TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting the metals and trivalent chromium data is numbered JC18361A, while the data package for the hexavalent chromium analyses is numbered JC18361. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC18361 together with the supplemental total organic carbon (TOC), sulfide screen and ferrous iron. The data for the

five target metals (antimony, chromium, nickel, thallium, and vanadium) were validated in this DV report, as were the hexavalent chromium, TOC, sulfide screen and ferrous iron data. The Cr+3 results were calculated as the difference between the total chromium and Cr+6 concentrations.

Data Review

Data, as presented in the analytical data packages SDG JC18361A and JC18361, was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012);
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC18361A

The data validation of the metals analytical data in SDG JC18361A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 7 post-excavation soil samples and one field blank were analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. The Cr+3 results were calculated as the difference between the total chromium and Cr+6 concentrations. Of the sample metals results detected in the 7 soil samples of SDG JC18361A, the nickel result in JC18361-6A was above the IGWSSL of 48 milligrams per kilogram (mg/kg), while the vanadium result in JC18361-5A exhibited a concentration above the SRS of 78 mg/kg for vanadium.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP93153 indicating possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution result for antimony was outside control limits in QC Batch MP93153, and nickel and thallium were outside control limits in QC Batch MP93135 associated with the field blank. However, the percent difference (%D) results were acceptable due to low initial sample concentrations (< 50 times instrument detection limit [IDL]). The serial dilution for chromium, nickel and vanadium in QC Batch MP93153 indicates possible matrix interference. The detection limits for antimony, chromium and thallium are elevated in JC18361-6A due to dilution required for high interfering element. All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample and field blank analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

The exceptions consisted of the 170% recovery of antimony and 150% recovery of chromium in CRID1 at 10:47 in analytical sequence MA39192 associated with the samples and the field blank.

However, the soil sample results were not subject to qualification because antimony was not detected in any of the soil samples and the antimony results are not subject to qualification because there is no positive bias in a non-detect result. Additionally, the chromium soil sample results are also not subject to qualification, because the detected chromium concentration in each of the soil samples was considerably above the affected range of 0 – 0.5 mg/kg (as extrapolated from another PPG SDG displaying a full metals data deliverable) in the associated soil samples.

Consequently, no soil sample or field blank results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks, the continuing calibration blanks (CCBs) or the field blank at the stated reporting limits, except for the 2.2 micrograms per liter (µg/L) thallium in CCB1 at 10:34 in analytical sequence MA39192. However, no samples were associated with this CCB, since all samples were analyzed after CCB2, which was acceptable with no detected target analytes. Hence, no soil sample results warranted qualification for any associated QC blank contamination in SDG JC18361A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for PPG QC batch sample JC18361-2A, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results of QC Batch GP93153 fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93153 Ω	JC18361-2A	Antimony	44.3 %	44.1 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ω – The samples associated with QC Batch MP93153 consist of JC18361-2A through -8A (inclusive).						

The antimony results in the seven affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 5.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 0 – 1.3%RPD for soil samples and 1.0 – 1.5 %RPD for the batch QC sample associated with the field blank analysis with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 96.5% - 103.5% for the soil sample metals analysis, and 99.5 – 105.5% for the aqueous matrix.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative stated that the RPD serial dilution result for antimony in QC Batch MP93153 and nickel and thallium in QC Batch MP93135 were outside control limits. However, the percent difference (%D) results were acceptable due to low initial sample concentrations (< 50 times IDL).

The case narrative also identified the serial dilution results being outside QC limits for chromium, nickel and vanadium in QC Batch MP93153 and stated that the differences indicate possible matrix interferences. Thus, the affected chromium, nickel and vanadium results in the samples associated with these elevated %D exceedances are subject to qualification (“EJ”) following the DV review, as discussed below. These QC results are detailed in Table 3 below.

Table 3. Serial Dilution Results Above QC Limits

QC Batch	QC Sample	Analyte	% Difference	DV Qualifier
MP93153 Ω	JC18361-2A	Chromium	17.0 %D	EJ
MP93153 Ω	JC18361-2A	Nickel	12.8 %D	EJ
MP93153 Ω	JC18361-2A	Vanadium	15.5 %D	EJ

Notes:
 EJ – The reported value is estimated because of the presence of interference; indeterminate bias direction.
 Ω – The samples associated with QC Batch MP93153 consist of JC18361-2A through -8A (inclusive).

The associated chromium, nickel, and vanadium results in samples with laboratory sample ID numbers ranging JB18361-2A through -8A, are qualified as estimated values and flagged with “EJ” to indicate that the result is an estimated value possibly experiencing variability in the reported value due to the presence of an interference in the sample matrix. The individual qualified results are presented in the summary table, Table 5.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

Sample JC18361-6A required a five-fold (5×) dilution for antimony, chromium and thallium analysis due to the presence of an interfering element, such that the antimony and thallium reporting limits for this sample were raised to values of < 11 and < 5.6 mg/kg, values above the respective IGWSSLs of 6 and 3 mg/kg, as detailed below in Table 4. Because chromium was detected (567 mg/kg) in this sample, the reporting limit is not affected by the dilution.

Table 4. Sample Reporting Limits Affected by Sample Dilution

Sample ID	Lab ID	Analyte	Reporting Limit (mg/kg)	Dilution Factor	Adjusted Result (mg/kg)	Remediation Standard (mg/kg)
PPG174 BERM SW03	JC18361-6A	Antimony	< 2.2	5	< 11	6
PPG174 BERM SW03	JC18361-6A	Thallium	< 1.12	5	< 5.6	3

Units – mg/kg (milligrams per kilogram)
 < - The analyte was analyzed for but was not detected above the stated reporting limit.

The interpretation of the reporting limits for antimony and thallium in JC18361-6A was not compromised because the nickel concentration was above the IGWSSL and the sample would need to be addressed in either additional review or some type of remedial action. Additionally, interpretation of the soil results was not compromised by this 5× dilution, and because thallium has not been detected in any of the soil samples during this phase of sampling at PPG.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC18361A were found to be compliant with the analytical methods for the analysis of metals in the 7 soil samples and one field blank using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP93153 associated with the 7 soil samples: JC18361-2A through JC18361-8A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 5.

Aside from the low MS/MSD recoveries for antimony, QC criteria were met for the ICP target analyte analyses, except for the serial dilution analysis for chromium, nickel, and vanadium in the QC batch associated with the 7 soil samples of this SDG, as summarized in Table 3 above and detailed below in Table 5. The chromium, nickel, and vanadium results in these samples are qualified as estimated values (flagged “EJ”) in the associated soil samples due to a potential variability in the reported results due to the possible presence of interference in the sample matrix with an indeterminate bias.

Table 5. Summary of Qualified Sample Metals Results in SDG JC18361A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174 BERM B01	JC18361-2A	Antimony	< 2.2	NJ-
PPG174 BERM B01	JC18361-2A	Chromium	26.1	EJ
PPG174 BERM B01	JC18361-2A	Nickel	24.1	EJ
PPG174 BERM B01	JC18361-2A	Vanadium	34.1	EJ
PPG174 BERM B01	JC18361-2A	Cr+3	25.5	J
PPG174 BERM B02	JC18361-3A	Antimony	< 2.2	NJ-
PPG174 BERM B02	JC18361-3A	Chromium	21.0	EJ
PPG174 BERM B02	JC18361-3A	Nickel	22.8	EJ
PPG174 BERM B02	JC18361-3A	Vanadium	28.8	EJ
PPG174 BERM B02	JC18361-3A	Cr+3	19.8	J
PPG174 BERM SW01	JC18361-4A	Antimony	< 2.5	NJ-
PPG174 BERM SW01	JC18361-4A	Chromium	47.9	EJ
PPG174 BERM SW01	JC18361-4A	Nickel	27.8	EJ
PPG174 BERM SW01	JC18361-4A	Vanadium	39.8	EJ
PPG174 BERM SW01	JC18361-4A	Cr+3	47.9	J
PPG174 BERM SW02	JC18361-5A	Antimony	< 2.2	NJ-
PPG174 BERM SW02	JC18361-5A	Chromium	21.7	EJ
PPG174 BERM SW02	JC18361-5A	Nickel	33.6	EJ
PPG174 BERM SW02	JC18361-5A	Vanadium	95.4	EJ
PPG174 BERM SW02	JC18361-5A	Cr+3	20.6	J
PPG174 BERM SW03	JC18361-6A	Antimony	< 11	NJ-
PPG174 BERM SW03	JC18361-6A	Chromium	567	EJ
PPG174 BERM SW03	JC18361-6A	Nickel	605	EJ

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174_BERM_SW03	JC18361-6A	Vanadium	51.0	EJ
PPG174_BERM_SW03	JC18361-6A	Cr+3	566	J
PPG174_BERM_SW04	JC18361-7A	Antimony	< 2.1	NJ-
PPG174_BERM_SW04	JC18361-7A	Chromium	95.8	EJ
PPG174_BERM_SW04	JC18361-7A	Nickel	19.2	EJ
PPG174_BERM_SW04	JC18361-7A	Vanadium	29.2	EJ
PPG174_BERM_SW04	JC18361-7A	Cr+3	82.9	J
PPG174_BERM_SW05	JC18361-8A	Antimony	< 2.3	NJ-
PPG174_BERM_SW05	JC18361-8A	Chromium	181	EJ
PPG174_BERM_SW05	JC18361-8A	Nickel	38.8	EJ
PPG174_BERM_SW05	JC18361-8A	Vanadium	61.0	EJ
PPG174_BERM_SW05	JC18361-8A	Cr+3	175	J

Key:

mg/kg - milligrams per kilogram

< –The analyte was analyzed for but was not detected above the stated reporting limit.

EJ –The reported value is estimated because of the presence of interference; indeterminate bias direction.

J – The result is an estimated value;

NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.

Because the total chromium results are qualified as estimated values (“EJ”), as are the Cr+6 results (“NJ-”), and the Cr+3 results are calculated from these two entities, it was judged appropriate to also qualify the Cr+3 results as estimated values (“J”).

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC18361

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the seven post-excavation soil samples and one QC batch for the field blank. The soil samples were re-analyzed in a third QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was detected in 6 of the 7 soil samples analyzed in SDG JC18361, with all sample Cr+6 results less than 13 mg/kg, all values below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery in QC Batch GP96946 was outside control limits indicating possible matrix interference. The soluble matrix spike recovery was also outside control limits, as were the post spike and pH-adjusted post spike recoveries in reanalysis QC Batch GP97000. There was good agreement between the sample and 1:5 dilution in the re-analysis. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibrations demonstrated acceptable correlation coefficients (“r”) with values of 0.99965 for the soil samples analysis, as well as 0.99980 for the aqueous fraction, values greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 100.0% to 102.9% for the QC batch associated with the analysis of 7 soil samples, and 104.5 and 105.4% for the aqueous fraction, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg), the continuing calibration blanks, or the field blank (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery were below the QC limits of 75-125% for QC Batch GP96946 associated with the 7 soil samples of this SDG, as presented below in Table 6. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP96946 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. All remaining MS recoveries were within QC limits.

Table 6. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC18361

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP96946 ¥	JC18361-2	Cr ⁺⁶ , soluble	66.4 %	NJ-	Low
GP96946 ¥	JC18361-2	Cr ⁺⁶ , insoluble	105.3 %	----	----
GP96946 ¥	JC18361-2	Cr ⁺⁶ , post-digestion spike	90 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
MS – Matrix spike
Cr⁺⁶ – Hexavalent chromium
NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
¥ – The samples associated with QC Batch GP96946 consist of JC18361-2 through -8 (inclusive).

The Cr+6 results qualified for low spike recoveries are flagged with “NJ-” (NJDEP, 2009; US EPA, 2010), as tabulated below in Table 8, together with the qualified results from the re-analysis of this QC batch.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from sample JC18361-2 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-BERM_B01) was 1.6%RPD, a value below the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (5.0%RPD) and pH (1.6%RPD)

also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 90.3% and 92.9% associated with the soil samples and 106.7% for the aqueous matrix, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method. The note on the MS Results Summary page indicated that there was good agreement between the sample and the 1:5 dilution in the reanalysis QC batch.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)
 C = Wet weight of sample (Kg)
 D = % Solids/100
 E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-BERM_SW04 (JC18361-7) was listed as 12.9 mg/kg on the reporting form and 0.2800 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.2800 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00242 \text{ Kg} \times 90.0/100} = \frac{0.02800}{0.002178} = 12.8558 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 12.9 \text{ mg/kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 12.9 mg/kg for Sample PPG174-BERM_SW04 was correctly reported. This was the highest detected Cr+6 concentration of the 6 detected results for the 7 soil samples of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Six of the seven soil samples were observed to fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a “reducing” soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions, while Cr+6 may increase under “oxidizing” conditions, provided there is a significant concentration of chromium available. The sample Cr+6 concentrations detected in the six (of seven analyzed) samples are all below 13 mg/kg, with total chromium concentrations for the samples with detected Cr+6 all less than 600 mg/kg. The detected result in JC18361-7 fell on or slightly above the Eh-pH phase diagram line suggesting a potential “oxidizing” soil character with a corresponding total chromium result of 95.8 mg/kg but is not expected to increase to levels approaching the SCC of 20 mg/kg, because of the relatively low total chromium concentration available to be oxidized.

It has been observed that PPG soil samples with total chromium concentrations less than 600 mg/kg do not exhibit Cr+6 concentrations that would increase to any significant degree that approach the SCC of 20 mg/kg for samples exhibiting “reducing” conditions. With few exceptions, PPG soil samples representing “oxidizing” conditions have been observed to exhibit chromium to Cr+6 ratios (Cr:Cr+6) ranging from approximately 6 to 75, or greater.

Summary for Hexavalent Chromium Analysis – SDG JC18361

Because the soluble MS recovery was below QC limits in the QC batch, the samples required reanalysis. The resultant data for the re-analysis batch consisting of 7 soil samples are summarized in the section below labelled “Cr+6 Re-analysis in SDG 18361.”

Cr+6 Re-analyses in SDG JC18361

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the re-analysis batch consisting of 7 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC19361-2R through -8R in QC Batch GP97000, including the calibrations ($r = 0.99990$, 96.8 – 97.3% CCV Recoveries), duplicate analysis (15.7%RPD), QC blanks, and blank spike analysis (86.3% – 101.7%). The soluble MS recovery was lower in the reanalysis, falling to 51% and still below QC limits, while the post-digestion spike and pH-adjusted post spikes also fell below QC limits, but the insoluble spike was still within QC limits, as detailed below. There was a good agreement between the samples and the 1:5 dilution.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the MS recoveries in QC Sample JC18361-2R were all slightly lower compared to the initial analysis, as observed below in Table 7. The insoluble MS recovery in JC18361-2R was still within the 75-125% QC limits.

Table 7. Hexavalent Chromium Re-analysis MS Recovery Results – JC18361

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97000 X	JC18361-2R	Cr ⁺⁶ , soluble	51.1 %	NJ-	Low
GP97000 X	JC18361-2R	Cr ⁺⁶ , insoluble	85.4 %	----	----
GP97000 X	JC18361-2R	Cr ⁺⁶ , post-digestion spike	80 %	NJ-	Low
GP97000 X	JC18361-2R	Cr ⁺⁶ , pH-adjusted post spike	81 %	NJ-	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. X – The samples associated with QC Batch GP97000 consist of JC18361-2R through -7R (inclusive).					

Since the soluble MS recovery in QC Batch GP97000 was still below the QC limits (75-125%), the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” because the soluble MS recovery was between 50 – 75% where DV guidelines recommend qualification of associated sample results as estimated values (NJDEP, 2009) for a potential low bias in the ability to recover Cr+6 in this QC batch. The qualified Cr+6 results of the reanalysis are presented below in Table 8 together with the results of the initial Cr+6 results.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC18361-2RT (PPG174_BERM_B01), a QC sample which was analyzed twice with detected concentrations of 0.64 and 0.94 mg/kg for both analyses, values well below the SCC of 20 mg/kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. The total organic carbon (TOC) analysis was performed within the 14-day analytical holding time. In accordance with the method, these analyses were performed on the sample experiencing the low spike recoveries. A concentration of total organic carbon (44,500 mg/kg) and the ferrous iron (Fe+2) with a result of 0.25 % were detected in the QC sample in JC18361-2RT, thereby indicating the likely presence of a reducing soil matrix in the soil sample, as suggested by the presence of this soil sample below the Eh-pH phase line, as are the five of the other six soil samples of this SDG.

The “reducing” conditions in the soil matrix appear supported by the detected TOC concentration and the detected Fe⁺² data in support of the results of the Eh-pH analyses.

Summary for Hexavalent Chromium Analysis – SDGs JC18361

The qualified soil sample results from the initial Cr+6 analysis in SDG JC18361 are presented below in Table 8 alongside those qualified results obtained from the reanalysis of the samples. Both sets of analytical Cr+6 results for samples JC18361-2 through -8 and their reanalysis are still both qualified as estimated values (NJ-) due to a potential low bias, although the soluble MS recoveries of the second analysis exhibited lower recoveries in the re-analyses that were performed within the 30-day holding time. The Cr+6 concentrations determined during the re-analysis of samples in SDG JC18361 differ slightly from those of the initial analysis, but all are still considerably below the SCC of 20 mg/kg.

Table 8. Comparison of Qualified Cr+6 Results in JC18361 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC18361 Result (mg/kg)	DV Qualifier	JC18361-R Results (mg/kg)	DV Qualifier
PPG174_BERM_B01	JC18361-2	Cr+6	0.64	NJ-	0.94	NJ-
PPG174_BERM_B02	JC18361-3	Cr+6	1.2	NJ-	1.1	NJ-
PPG174_BERM_SW01	JC18361-4	Cr+6	< 0.51	NJ-	1.3	NJ-
PPG174_BERM_SW02	JC18361-5	Cr+6	1.1	NJ-	0.91	NJ-
PPG174_BERM_SW03	JC18361-6	Cr+6	0.70	NJ-	1.2	NJ-
PPG174_BERM_SW04	JC18361-7	Cr+6	12.9	NJ-	10.5	NJ-
PPG174_BERM_SW05	JC18361-8	Cr+6	6.2	NJ-	5.1	NJ-
<p>< –The analyte was analyzed for but was not detected above the stated reporting limit. NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.</p>						

Since the soluble MS recoveries fell between 50 – 75%, the Cr+6 results in both the initial analysis and the re-analysis are qualified as estimated values and flagged with NJ- in accordance with DV guidance (NJDEP, 2009).

Although the samples were re-analyzed within the 30-day holding time, the Cr+6 concentrations differed only slightly upon reanalysis. Despite the decrease in the spike recovery values in the reanalysis, the changes in Cr+6 concentrations were mixed, with some results increasing and others lower than initially detected. This may be due primarily to sample non-homogeneity. Regardless, all Cr+6 sample results exhibited a Cr+6 values considerably below the SCC of 20 mg/kg, consistent with the redox state of the sample’s soil environment.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
EJ	The reported value is estimated because of the presence of interference; indeterminate bias direction.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

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US EPA, CLP, 2014, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9335.0-131, EPA-540-R-13-001, August 2014.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC18361/JC18361A

1. Were the appropriate sample preservation requirements met?..... Yes No

2. Were appropriate sample holding times (for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Sample JC18361-6A was diluted 5x for antimony, chromium and thallium due to the presence of a high interfering element.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

The reporting limits for antimony and thallium in Sample JC18361-6A exceeded the respective IGWSSL limits of 6 and 3 mg/kg.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The nickel result in JC18361-6A and vanadium in Sample JC18361-5A exceeded the IGWSSL of 48 mg/kg and SRS of 78 mg/kg, respectively.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... Yes **No**
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2, 3, 6, and 7 for QC details. Qualified sample results are presented in Tables 5 and 8 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC18699/JC18699A/JC18699R
Sample Dates: April 20, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Trivalent chromium, Method 6010C/7196A M
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Reviewer: Janis V. Giga. Ph.D., REP5554
Report Date: May 24, 2016

This data validation (DV) report presents the data review and result qualifications for four (4) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on April 20, 2016, for sample delivery group (SDG) JC18699, as well as JC18699A and JC18699R. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC18699A, JC18699 and JC18699R were found to be compliant with the analytical methods employed for the analysis of metals, hexavalent chromium (Cr+6), and trivalent chromium (Cr+3) in the 4 collected post-excavation soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC18699-1A through JC18699-4A (inclusive)
- Chromium, nickel and vanadium ("EJ") in Samples JC18699-1A through JC18699-4A (inclusive)
- Trivalent chromium ("J") in Samples JC18699-1A through JC18699-4A (inclusive)
- Hexavalent chromium ("*NJ-") in Samples JC18699-1 through JC18699-4 (inclusive)
- Hexavalent chromium ("NJ-") in reanalysis samples JC18699-1R through JC18699-4R (inclusive)

No other sample results in SDG JC18699A, JC18699, and JC18699R required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except nickel in samples JC18699-1A, -3A, and -4A, while the hexavalent chromium (Cr+6) concentrations in JC18699-1 and -2 were above the Soil Cleanup Criterion (SCC) of 20 mg/kg in the respective SDGs, as were the re-analysis results for JB18699-1R and -2R. A data validation checklist is provided in Attachment A to summarize the observations during the DV

review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Tables 4 and 8 of this DV report.

Sample Receipt

The four (4) soil samples collected April 20, 2016, were received intact and appropriately preserved the same day, April 20, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 2.9 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC18699A and JC18699

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174 FCC	JC18699-1A	4/20/2016	Soil	Metals, Cr+3
PPG174 RR_B01	JC18699-2A	4/20/2016	Soil	Metals, Cr+3
PPG174 RR_SW01	JC18699-3A	4/20/2016	Soil	Metals, Cr+3
PPG174 RR_SW02	JC18699-4A	4/20/2016	Soil	Metals, Cr+3
PPG174 FCC	JC18699-1	4/20/2016	Soil	Cr+6
PPG174 RR_B01	JC18699-2	4/20/2016	Soil	Cr+6
PPG174 RR_SW01	JC18699-3	4/20/2016	Soil	Cr+6
PPG174 RR_SW02	JC18699-4	4/20/2016	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. Cr+3 – Trivalent chromium calculated as (Chromium – Cr+6) from Methods 6010C and 7196A.				

The data package presenting the metals and trivalent chromium data is numbered JC18699A, while the data package for the hexavalent chromium analyses is numbered JC18699. The data for the re-analysis of the samples for hexavalent chromium data are found in JC18699R. The Cr+3 results were calculated as the difference between the total chromium and initial Cr+6 concentrations.

Data Review

Data, as presented in the analytical data packages SDG JC18699A, JC18699, and JC18699R was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).

- US EPA “ICP-AES Data Validation, SOP No. HW-2a, Revision 15” (USEPA, 2012).
- NJDEP Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC18699A

The data validation of the metals analytical data in SDG JC18699A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The four post-excavation soil samples were analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. The Cr+3 results were calculated as the difference between the total chromium and Cr+6 concentrations. Of the sample metals results detected in the 4 soil samples of SDG

JC18669A, the nickel results in JC18699-1A, -3A, and -4A were above the IGWSSL of 48 milligrams per kilogram (mg/kg).

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP93153 indicating possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution result for antimony was outside control limits in QC Batch MP93153, however, the percent difference (%D) result was acceptable due to a low initial sample antimony concentration (< 50 times instrument detection limit [IDL]). The serial dilution for chromium, nickel and vanadium indicates possible matrix interference. All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and "low calibration check standard" ("CRI" standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

One exception consisted of the 170% recovery of antimony and 150% recovery of chromium in CRID1 at 10:47 in analytical sequence MA39192 associated with only the QC samples, while the antimony recovery of 143.3% was observed in the closing CRID2. No soil samples were associated with the contract required detection limit (CRDL) standards of this analytical sequence.

Another exception consisted of the 0% recovery of thallium in CRID2 at 14:55 in analytical sequence MA39236 associated with the four soil samples. However, the soil sample results were not affected because the reporting limits for the non-detect thallium results are above the respective affected range where results may be subject to qualification. The affected range is approximately 0 – 0.44 mg/kg for thallium, where the corresponding reporting limits of approximately 1 mg/kg are above the affected range of thallium and sample results are, hence, not subject to qualification.

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), except thallium in CCB1 at 10:34 in analytical sequence MA39192 containing QC samples. However, because CCB1 was not directly associated with these QC samples, which were analyzed later in the sequence, and thallium was not detected in any soil samples of this SDG, no soil sample results warranted qualification for any associated QC blank contamination in SDG JC18699A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis
(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for the PPG QC batch sample JC18361-2A from SDG JC18361A, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results of QC Batch GP93153 fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93153 Ω	JC18361-2A	Antimony	44.3 %	44.1 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias. Ω – The samples associated with QC Batch MP93153 consist of JC18669-1A through -4A (inclusive).						

The antimony results in the four affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 4.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 0 – 1.3%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 87.4% - 92.9% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative stated that the RPD serial dilution result for antimony was outside control limits in QC Batch MP93153; however, the percent difference (%D) result was acceptable due to a low initial sample antimony concentration (< 50 times IDL).

The case narrative identified the serial dilution results being outside QC limits for chromium, nickel and vanadium in QC Batch MP93153 and stated that the differences indicate possible matrix interferences. Thus, the affected chromium, nickel and vanadium results in the samples associated with these elevated %D exceedances are subject to qualification as estimated values flagged with “EJ” following the DV review, as discussed below. These QC results are detailed in Table 3 below.

Table 3. Serial Dilution Results Above QC Limits

QC Batch	QC Sample	Analyte	% Difference	DV Qualifier
MP93153 Ω	JC18361-2A	Chromium	17.0 %D	EJ
MP93153 Ω	JC18361-2A	Nickel	12.8 %D	EJ
MP93153 Ω	JC18361-2A	Vanadium	15.5 %D	EJ

Note:
EJ – The reported value is estimated because of the presence of interference; indeterminate bias direction.
Ω – The samples associated with QC Batch MP93153 consist of JC18699-1A through -4A (inclusive).

The associated chromium, nickel, and vanadium results in samples with laboratory sample ID numbers ranging JB18699-1A through -4A, are qualified as estimated values and flagged with “EJ” to indicate that the result is an estimated value possibly experiencing variability in the reported value due to the presence of an interference in the sample matrix. The individual qualified results are presented in the summary table, Table 4.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC18699A were found to be compliant with the analytical methods for the analysis of metals in the 4 soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP93153 associated with the 4 soil samples: JC18699-1A through JC18699-4A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 4.

Aside from the low MS/MSD recoveries for antimony, QC criteria were met for the ICP target analyte analyses, except for the serial dilution analysis for chromium, nickel, and vanadium in the QC batch associated with the 4 soil samples of this SDG, as summarized in Table 3 above, and detailed below in Table 4. The chromium, nickel, and vanadium results in these samples are qualified as estimated values (flagged “EJ”) in the associated soil samples due to a potential variability in the reported results due to the possible presence of interference in the sample matrix with an indeterminate bias.

Table 4. Summary of Qualified Sample Metals Results in SDG JC18699A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174 FCC	JC18699-1A	Antimony	< 2.1	NJ-
PPG174 FCC	JC18699-1A	Chromium	238	EJ

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174 FCC	JC18699-1A	Nickel	63.4	EJ
PPG174 FCC	JC18699-1A	Vanadium	53.1	EJ
PPG174 FCC	JC18699-1A	Cr+3	214	J
PPG174 RR B01	JC18699-2A	Antimony	< 2.1	NJ-
PPG174 RR B01	JC18699-2A	Chromium	196	EJ
PPG174 RR B01	JC18699-2A	Nickel	17.3	EJ
PPG174 RR B01	JC18699-2A	Vanadium	29.0	EJ
PPG174 RR B01	JC18699-2A	Cr+3	171	J
PPG174 RR SW01	JC18699-3A	Antimony	< 2.1	NJ-
PPG174 RR SW01	JC18699-3A	Chromium	169	EJ
PPG174 RR SW01	JC18699-3A	Nickel	52.8	EJ
PPG174 RR SW01	JC18699-3A	Vanadium	40.4	EJ
PPG174 RR SW01	JC18699-3A	Cr+3	162	J
PPG174 RR SW02	JC18699-4A	Antimony	< 2.2	NJ-
PPG174 RR SW02	JC18699-4A	Chromium	199	EJ
PPG174 RR SW02	JC18699-4A	Nickel	60.5	EJ
PPG174 RR SW02	JC18699-4A	Vanadium	48.3	EJ
PPG174 RR SW02	JC18699-4A	Cr+3	194	J

Key:

mg/kg - milligrams per kilogram

< -The analyte was analyzed for but was not detected above the stated reporting limit.

EJ -The reported value is estimated because of the presence of interference; indeterminate bias direction.

J - The result is an estimated value;

NJ- - The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.

Because the total chromium results are qualified as estimated values ("EJ"), as are the Cr+6 results ("NJ-") (See Table 8), and the Cr+3 results are calculated from these two estimated entities, it was judged appropriate to also qualify the Cr+3 results as estimated values ("J").

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC18699

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the four post-excavation soil samples. The soil samples were re-analyzed in a second QC batch whose data are presented in SDG JC18699R.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was detected in each of the four soil samples analyzed in SDG JC18699, with sample Cr+6 results exceeding the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg in samples JC18699-1 and -2.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery in QC Batch GP97079 was outside control limits indicating possible matrix interference. The RPD result for the duplicate analysis was outside control limits due to possible sample non-homogeneity. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with value of 0.99993 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 94.1% to 94.3% for the QC batch associated with the analysis of 4 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP97079 associated with the 4 soil samples of this SDG, as presented below in Table 5. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP97079 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. All remaining MS recoveries were within QC limits.

Table 5. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC18699

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97079 ¥	JC18699-3	Cr ⁺⁶ , soluble	63.6 %	NJ-	Low
GP97079 ¥	JC18699-3	Cr ⁺⁶ , insoluble	100.8 %	----	----
GP97079 ¥	JC18699-3	Cr ⁺⁶ , post-digestion spike	110 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. ¥ – The samples associated with QC Batch GP97079 consist of JC18699-1 through -4 (inclusive).					

The Cr+6 results qualified for low spike recoveries are flagged with “NJ-” (NJDEP, 2009; US EPA, 2010), as tabulated below in Table 8, together with the qualified results from the re-analysis of this QC batch. The Cr+6 results in the 4 soil samples in are qualified as estimated values because the soluble MS recovery was within the 50-75% QC range where DV guidelines recommend qualification of associated samples results to be flagged with “J” due to a potential low bias (NJDEP, 2009).

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from Sample JC18699-3. The difference between the duplicate soil sample aliquots for Cr+6 in soil this sample (PPG174_RR_SW01) was 76.7%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD QC limit for soil samples (US EPA, 2010; AECOM, 2010), as presented below in Table 6. A possible cause of the observed differences between the duplicate results may be attributable to sample non-homogeneity. The %RPD values for redox potential (3.3%RPD) and pH (0.4%RPD) displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was above the QC limit for soil samples, the associated sample results are qualified as estimated values with an indeterminate bias and are to be flagged with “*J” to indicate that there is potential variability in the analytical precision.

Table 6. Duplicate Analysis Results Outside QC Limits

QC Batch	QC Sample	Analyte	Original Result (mg/kg)	Duplicate (mg/kg)	Difference	DV Qualifier
GP97079 ¥	JC18699-3	Cr+6	7.0	15.7	76.7 %RPD	*J
mg/kg - milligrams per kilogram QC Limit is 35%RPD; * - Duplicate analysis not within control limits; indeterminate bias direction. J - The reported result is an estimated value. ¥ - The samples associated with QC Batch GP97079 consist of JC18699-1 through -4 (inclusive).						

Since the duplicate analysis for Cr+6 had exceeded the QC limit for duplicate soil sample analysis, the associated PPG samples with laboratory ID numbers JC18699-1 through -4 (inclusive) were qualified as estimated values due to the potential variability in the analytical precision. Because the soluble MS recovery was also below QC limits, the Cr+6 results for these four soil samples are flagged with “*NJ-” in the summary table below, Table 8.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 92.0% and 94.4% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)
 C = Wet weight of sample (kg)
 D = % Solids/100
 E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174_RR_B01 (JC18699-2) was listed as 24.7 mg/kg on the reporting form and 0.5485 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.5485 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00247 \text{ Kg} \times 90.0/100} = \frac{0.05485}{0.0022230} = 24.6739 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 24.7 \text{ mg/kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 24.7 mg/kg for Sample PPG174_RR_B01 was correctly reported. This was the highest detected Cr+6 concentration of the four detected results for the 4 soil samples of this SDG, a value above the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

All four soil samples were observed to fall above the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of an “oxidizing” soil environment. The Cr+6 concentration may increase under “oxidizing” conditions, provided there is a significant concentration of chromium available. It has been observed at various PPG sampling locations that even for soils in an oxidizing condition, the Cr:Cr+6 ratio may range from 7 to 75, while samples under “reducing” conditions typically exhibit ratios of approximately 20 or greater.

Summary for Hexavalent Chromium Analysis – SDG JC18699

Since the soluble MS spike recovery of 63.6% was below QC limits in the QC sample of QC Batch GP97079, the soil sample in this QC batch required reanalysis. The remaining QC results associated with the hexavalent chromium analysis were within QC limits, except for the duplicate analysis which led to qualification of the Cr+6 as estimated values flagged with “*NJ-”. Therefore, the Cr+6 result for the samples of SDG JC18699 were qualified following the DV review and flagged with “*NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix and possible variability in the analytical precision. Consequently, the soil samples of this QC Batch GP97079 were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analyses in SDG JC18699R.”

Cr+6 Re-analyses in SDG JC18699R

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the batch consisting of 4 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC18699-1R through -4R in QC Batch GP97119, including the calibrations ($r = 0.99995$, 93.8 – 94.0% CCV Recoveries), QC blanks, duplicate analysis (≤ 35 %RPD), and blank spike analysis (87.8% and 90.8%). The soluble MS recovery was slightly higher in the reanalysis, while the post spike was lower, but still within QC limits, as detailed below.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the MS recoveries in QC Sample JC18699-3R were all slightly different compared to the initial analyses, as observed below in Table 7. The insoluble MS recovery in JC18699-3R was still well within the 75-125% QC limits.

Table 7. Hexavalent Chromium Re-analysis MS Recovery Results – JC18699R

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97119 £	JC18699-3R	Cr ⁺⁶ , soluble	68.8 %	NJ-	Low
GP97119 £	JC18699-3R	Cr ⁺⁶ , insoluble	97.1 %	----	----
GP97119 £	JC18699-3R	Cr ⁺⁶ , post-digestion spike	89 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
MS – Matrix spike
Cr⁺⁶ – Hexavalent chromium
NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
£ –The samples associated with QC Batch GP97119 consist of JC18699-1R through -4R (inclusive).

Since the soluble MS recovery in QC Batch GP97119 was still below the QC limits (75-125%), the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” for a potential low bias in the ability to recover Cr+6 in this QC batch. The qualified Cr+6 results of the reanalysis are presented below in Table 8 together with the results of the initial Cr+6 results.

Duplicate Sample Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots. The differences between the duplicate soil sample aliquot concentrations for Cr+6 in the sample aliquots was listed as 33.3%RPD. Although this RPD value exceeded the 20%RPD laboratory QC limit, it was below the DV advisory QC limit of 35%RPD for technical review purposes of soil data (USEPA, 2010), thereby meeting QC requirements and avoiding the need to qualify the Cr+6 results from the reanalysis. Hence, the Cr+6 results in the associated samples were not qualified for the duplicate analysis result and analytical precision is considered acceptable in the re-analysis.

Summary for Hexavalent Chromium Analysis – SDGs JC18699 and JC18699R

The qualified soil sample results from the initial Cr+6 analysis in SDG JC18699 are presented below in Table 8 alongside those qualified results obtained from the reanalysis of the samples from SDG JC18699R. Both sets of analytical Cr+6 results for samples JC18669-1 through -4 and their reanalysis are still both qualified as estimated values (NJ-) due to a potential low bias, although the soluble MS recovery of the second analysis exhibited a slightly better recovery in the re-analysis

that was performed within the 30-day holding time. The Cr+6 concentrations determined during the re-analysis of samples in SDG JC18699R differ slightly from those of the initial analysis but are quite similar. The samples with Cr+6 concentrations exceeding the SCC of 20 mg/kg in the initial analysis also exceeded the SCC in the re-analysis by a similar amount.

Table 8. Comparison of Qualified Cr+6 Results in JC18699 and Re-analysis in JC18699R

Client ID	Laboratory Sample ID	Analyte	JC18699 Result (mg/kg)	DV Qualifier	JC18699-R Results (mg/kg)	DV Qualifier
PPG174 FCC	JC18699-1	Cr+6	24.4	*NJ-	25.4	NJ-
PPG174 RR B01	JC18699-2	Cr+6	24.7	*NJ-	24.5	NJ-
PPG174 RR SW01	JC18699-3	Cr+6	7.0	*NJ-	8.5	NJ-
PPG174 RR SW02	JC18699-4	Cr+6	4.9	*NJ-	6.4	NJ-

mg/kg - milligrams per kilogram
 < -The analyte was analyzed for but was not detected above the stated reporting limit.
 * - Duplicate analysis not within control limits; indeterminate bias direction.
 J - The reported result is an estimated value.
 NJ- - The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

Since the soluble MS recoveries fell between 50 – 75% in the initial analysis and re-analysis, the Cr+6 results in both the initial analysis and the re-analysis are qualified as estimated values and flagged with “*NJ-” and “NJ-”, respectively, in accordance with DV guidance (NJDEP, 2009) due to a potential low bias in the ability to recover Cr+6 from the sample matrix.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
*	Duplicate analysis not within control limits; indeterminate bias direction.
EJ	The reported value is estimated because of the presence of interference; indeterminate bias direction.
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

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New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC18699/JC18699A/JC18699R

- 1. Were the appropriate sample preservation requirements met?..... Yes No
- 2. Were appropriate sample holding times (for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.
- 3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.
- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.
- 5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The nickel results in Samples JC19699-1A, -3A, and -4A exceeded the IGWSSL of 48 mg/kg. The Cr+6 results in samples JC18699-1 and -2, and re-analysis samples JC18699-1R and -2R, exceeded the SCC of 20 mg/kg.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.
- 7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No

9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**

If "yes", please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds
of concern at the site met? Yes **No**

12. Were the QC Summary Forms reviewed?..... **Yes** No

13. Internal Standards acceptable..... **Yes** No

14. MS/MSD acceptable..... Yes **No**

15. Calibration summaries acceptable..... **Yes** No

16. Serial dilutions acceptable..... Yes **No**

17. Inorganic duplicates acceptable..... Yes **No**

18. LCS recovery acceptable..... **Yes** No

19. Other QC acceptable?..... **Yes** No

Provide a brief explanation, if applicable.

Refer to DV report tables 2, 3, 5, 6, and 7 for QC details. Qualified sample results are presented in Tables 4 and 8 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC19001/JC19001A
Sample Dates: April 25, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: June 14, 2016

This data validation (DV) report presents the data review and result qualifications for four (4) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on April 25, 2016, for sample delivery group (SDG) JC19001, as well as JC19001A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC19001A and JC19001 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 4 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC19001-1A through JC19001-4A (inclusive)
- Chromium and nickel ("J") in Samples JC19001-2A and JC19001-4A
- Hexavalent chromium ("NJ-") in Samples JC19001-1 through JC19001-4 (inclusive)
- Hexavalent chromium ("NJ-") in reanalysis samples JC19001-1R through JC19001-4R (inclusive)

No other sample results in SDG JC19001A and JC19001 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except the nickel results in Samples JC19001-3A and JC19001-4A, while the hexavalent chromium (Cr+6) concentrations were below the Soil Cleanup Criterion (SCC) in the respective SDGs, except for JC19001-3 and its re-analysis JC19001-3R. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Tables 3 and 7 of this DV report.

Sample Receipt

The four (4) post-excavation soil samples collected April 25, 2016, were received intact and preserved appropriately the same day, April 25, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 4.3 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC19001A and JC19001

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174 RR B01R	JC19001-1A	4/25/2016	Soil	Metals
PPG174 RR SW01R	JC19001-2A	4/25/2016	Soil	Metals
PPG174 FCCR	JC19001-3A	4/25/2016	Soil	Metals
PPG174 DUP01	JC19001-4A	4/25/2016	Soil	Metals
PPG174 RR B01R	JC19001-1	4/25/2016	Soil	Cr+6
PPG174 RR B01R	JC19001-1 RT	4/25/2016	Soil	TOC, SS, Fe2+
PPG174 RR SW01R	JC19001-2	4/25/2016	Soil	Cr+6
PPG174 FCCR	JC19001-3	4/25/2016	Soil	Cr+6
PPG174 DUP01	JC19001-4	4/25/2016	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting the metals data is numbered JC19001A, while the data package for the hexavalent chromium analyses is numbered JC19001. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC19001 together with the supplemental total organic carbon (TOC), sulfide screen and ferrous iron. The samples data were validated for the five target metals (antimony, chromium, nickel, thallium, and vanadium), as were the hexavalent chromium data, and supplemental TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC19001A and JC19001 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).

- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC19001A

The data validation of the metals analytical data in SDG JC19001A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|---------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | Field duplicate sample analysis |
| √ Data qualifiers | √ Data package completeness |

The 4 post-excavation soil samples were analyzed for the 5 target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 4 samples of SDG JC19001A, the nickel results in 2 samples (JC19001-3A and JC19001-4A) exhibited a concentration above the IGWSSL of 48 milligrams per kilogram (mg/kg) for nickel.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP93332 indicating possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution results for antimony and thallium were outside control limits in QC Batch MP93332; however, the percent difference (%D) results were acceptable due to a low initial sample concentrations (< 50 times instrument detection limit [IDL]). The detection limit for thallium is elevated in JC19001-4A due to dilution required for high interfering element. All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC19001A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for PPG Sample JC19001-1A in QC Batch MP93332, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results of QC Batch GP93332 fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93332 Ω	JC19001-1A	Antimony	61.2 %	63.5 %	NJ-	Low

QC Limits are 75-125%;
MS – Matrix spike
MSD – Matrix spike duplicate.
NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.
Ω – The samples associated with QC Batch MP93332 consist of JC19001-1A through -4A (inclusive).

The antimony results in the four affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in summary table, Table 4.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 2.7 – 7.6%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 101.5% - 106.1% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative also stated that the RPD serial dilution results for antimony and thallium were outside control limits in QC Batch MP93332; however, the percent difference (%D) results were acceptable due to a low initial sample concentrations (< 50 times IDL). The remaining analyte serial dilution results associated with the soil samples ranged from 1.2 – 4.5%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Field Duplicate Sample Analysis (QC Limit ≤ 50 %RPD)

One set of field duplicate samples was collected as part of SDG JC19001A. Field duplicate sample collection and analysis can provide a determination of sampling representativeness and precision. Gross differences between field sample duplicates can be an indication of inconsistent sampling techniques or sample matrix complexities/non-homogeneity.

The advisory data validation guidelines for field duplicate soil sample analysis vary. There is no NJDEP DV guideline for qualifying field duplicate results (NJDEP, 2002). Recently, EPA has recommended qualifying field duplicate results that differ by more than 50%RPD or $> 2 \times$ contract required quantification limit (CRQL) (USEPA, 2014), while the Field Sampling Plan for Hudson County chromium sites lists a data quality objective (DQO) of 50%RPD for soil samples (AECOM, 2010).

The results for the analysis of the one pair of field duplicate samples are presented below in Table 3. It is apparent that the results for chromium and nickel in the field duplicate samples of PPG174_RR_SW01R were dissimilar, with concentrations differing by more than 50%RPD, while the antimony and thallium results differed by less than two times the reporting limit value ($< 2 \times$ CRQL). Therefore, chromium and nickel results are subject to qualification as estimated concentrations. Soil sample non-homogeneity may have contributed to the observed disparities.

Table 3. Comparison of Field Duplicate Soil Sample Results – SDG JB19001A

Analyte	PPG174_RR_SW01R (mg/kg)	PPG174_DUP01 (mg/kg)	% RPD	DV Flag
Antimony	< 2.1 NJ-	< 2.1 NJ-	< 2 × CRQL	-
Chromium	140	295	71.3 %	J
Nickel	35.6	67.8	62.3 %	J
Thallium	< 1.1	< 2.1	< 2 × CRQL	-
Vanadium	36.6	44.5	19.5	-
Total Solids	92.3 %	91.7 %	0.6 %	-

mg/kg - milligrams per kilogram
 < – The analyte was not detected at the stated reporting limit;
 J – The reported result is an estimated value;
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
 CRQL – The value representing the US EPA CLP contract required quantitation limit, often represented by the reporting limit;
 < 2 × CRQL – The difference between field duplicate results was less than two times the CRQL and meets QC requirements for sampling representativeness.

Consequently, due to the disparity in the chromium and nickel results for the field duplicate samples PPG174_RR_SW01R and PPG174_DUP01, these results are subject to qualification and are to be flagged with “J”, as indicated in Table 3 above.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

The case narrative did identify that there was one sample (JC19001-4A) with a thallium reporting limit that was elevated due to the presence of “high interfering element.” Review of the data indicated that this reporting limit for thallium was below the respective IGWSSL limit of 3 mg/kg. No other samples were diluted in the metals analysis, such that all remaining reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The post-excavation soil sample analytical results for the samples of SDG JC19001A were found to be compliant with the analytical methods for the analysis of metals in the 4 post-excavation soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP93332 associated with the four soil samples: JC19001-1A through JC19001-4A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 4.

The chromium and nickel results for the field duplicate samples PPG174_RR_SW01R and PPG174_DUP01 are qualified as estimated values and flagged with “J” due to potential variability in sampling representativeness and precision.

Table 4. Summary of Qualified Sample Metals Results in SDG JC19001A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174 RR B01R	JC19001-1A	Antimony	2.3	NJ-
PPG174 RR SW01R	JC19001-2A	Antimony	< 2.1	NJ-
PPG174 RR SW01R	JC19001-2A	Chromium	140	J
PPG174 RR SW01R	JC19001-2A	Nickel	35.6	J
PPG174 FCCR	JC19001-3A	Antimony	< 2.0	NJ-
PPG174 DUP01	JC19001-4A	Antimony	< 2.1	NJ-
PPG174 DUP01	JC19001-4A	Chromium	295	J
PPG174 DUP01	JC19001-4A	Nickel	67.8	J

Key:
 mg/kg - milligrams per kilogram
 < - The analyte was analyzed for but was not detected above the stated reporting limit.
 J - The reported result is an estimated value.
 NJ- - The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC19001

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the four post-excavation soil samples. The soil samples were re-analyzed in a second QC batch in SDG JC19001.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- √ Data qualifiers
- Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Field duplicate sample analysis

Hexavalent chromium was detected in each of the four post-excavation soil samples analyzed in SDG JC19001, with sample Cr+6 results less than the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg, except for JC19001-3 and its re-analysis JC19001-3R.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike, post spike and pH adjusted post spike recoveries in QC Batch GP97198 were outside control limits, as well as in reanalysis QC Batch GP97260. The RPD value for the duplicate analysis in the analysis QC Batch GP97198 was above control limits, but the RPD was acceptable due to low duplicate and sample concentrations. There was not a good agreement between the sample and 1:5 dilution in both analyses. The ferrous iron and sulfide screen test were analyzed after completion of Cr+6 testing (outside of

normal hold time) in order to provide more information about the possible impact of the sample matrix on Cr+6 recoveries. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99997 for the soil samples analysis, as well as 0.99984 for the re-analysis, values greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 98.7% to 99.2% for the QC batch associated with the analysis of 4 soil samples, and 91.4 - 91.9% for the re-analysis, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP97198 associated with the 4 soil samples of this SDG, as well as below 50%, as presented below in Table 5. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP97198 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. The post-digestion and pH-adjusted post-digestion spike recoveries were also below the respective QC limits of 85-115%.

Table 5. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC19001

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97198 X	JC19001-1	Cr ⁺⁶ , soluble	28.5 %	NJ-	Low
GP97198 X	JC19001-1	Cr ⁺⁶ , insoluble	87.0 %	----	----
GP97198 X	JC19001-1	Cr ⁺⁶ , post-digestion spike	70.55 %	NJ-	Low
GP97198 X	JC19001-1	Cr+6, pH-adjusted post spike	70.46 %	NJ-	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias. X – The samples associated with QC Batch GP97198 consist of JC19001-1 through -4 (inclusive).					

The Cr+6 results qualified for low spike recoveries are flagged with “NJ-” (US EPA, 2012), as tabulated below in Table 8, together with the qualified results from the re-analysis of this QC batch. Qualification of these Cr+6 results is discussed in the Summary section at the end of the Cr+6 review. Sample Cr+6 results are subject to rejection, but were not, based on data usability concepts as explained in the conclusion summarizing the rationale for the qualifications.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from Sample location PPG174_RR_B01R (JC19001-1) for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample JC19001-1 was 22.2%RPD, a value above the 20%RPD laboratory QC limit, but within the 35%RPD DV advisory QC limit for technical review of soil sample data (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (1.3%RPD) and pH (0.1%RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 were below the QC limit for soil samples, the

associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 92.5% and 93.4% associated with the initial soil sample analysis, and 88.3% and 93.0% for the re-analysis, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as serial dilution is not a requirement of the analytical method and serial dilution is not addressed in DV guidelines (NJDEP, 2009). A 1:5 dilution was actually performed and there was poor agreement between the sample and the diluted aliquot. However, even though the results may be considered for qualification, the Cr+6 results are already qualified as estimated values and are flagged with "NJ-" for the low spike recoveries.

Field Duplicate Analysis (QC Limit ≤ 50%RPD)

The results for the analysis of one set of field duplicate samples are presented below in Table 6. The difference for the low-level concentrations observed in the field duplicate samples from sampling location PPG174_RR_SW01R differed by 27.3%RPD, which is below the QC limit of 50%RPD for soil samples (US EPA, 2012).

Table 6. Comparison of Field Duplicate Soil Sample Results – SDG JC19001

Analyte	PPG174_RR_SW01R (mg/kg)	PPG174_DUP01 (mg/kg)	% RPD	DV Flag
Hex.Chromium	5.7 NJ-	7.5 NJ-	27.3 %	----
mg/kg - milligrams per kilogram NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.				

Thus, the field duplicate results for the field duplicate samples from PPG174_RR_SW01R demonstrated acceptable field sampling representativeness and precision, with field duplicate soil sample results differing by less than 30%RPD. No soil sample Cr+6 results were qualified for sampling representativeness issues.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$Cr^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174_FCCR (JC19001-3) was listed as 53.9 mg/kg on the reporting form and 0.2695 mg/L on the quantitation report in the raw data for a 5-fold dilution. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.2695 \text{ mg/L} \times 0.1 \text{ L} \times 5}{0.00257 \text{ Kg} \times 97.3/100} = \frac{0.13475}{0.00250060} = 53.8868 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 53.9 \text{ mg/kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 53.9 mg/kg for Sample PPG174_FCCR was correctly reported. This was the highest detected Cr+6 concentration of the four detected results for the 4 soil samples of this SDG, a value clearly above the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Three of the four soil samples were observed to fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentrations are also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total chromium concentrations are all less than 300 mg/kg, thereby making it highly unlikely that Cr+6 concentrations would increase to any significant degree, as observed in many other PPG data packages with total chromium concentrations below 500 mg/kg.

Sample JC19001-3 was observed to fall above the Eh-pH phase line indicating that the soil matrix is considered to experience “oxidizing” soil conditions. Since this sample exhibited a Cr+6 concentration above the SCC of 20 mg/kg, the sample location will undergo further review and action.

Calculation of total chromium to Cr+6 ratios (Cr:Cr+6) for these four samples revealed that the three samples presumed to exhibit “reducing” soil conditions, not favorable for oxidizing chromium to Cr+6, ranged 25 to 40, while the sample under “oxidizing” conditions exhibited a ratio of 3.2, a value considerably lower than observed in many other oxidizing PPG soils, which tend to exhibit Cr:Cr+6 ratios above 10.

Hence, based on the sample total chromium and Cr+6 concentrations, it is highly unlikely that any of the affected samples in the “reducing” zone would approach the SCC for Cr+6 of 20 mg/kg due to limitation created by the relatively low total chromium concentrations available for potential oxidation.

Summary for Hexavalent Chromium Analysis – SDG JC19001

Since the soluble MS spike recovery of 28.5% was below QC limits in the QC sample of QC Batch GP97198, the soil sample in this QC batch required reanalysis. The Cr+6 results for the associated samples were qualified following the DV review and flagged with “NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix. Consequently, the soil samples of this QC Batch GP97198 were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analysis in JC19001”.

Cr+6 Re-analysis in SDG JC19001

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the re-analysis batch consisting of 4 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC19001-1R through -4R in QC Batch GP97260, including the calibrations (r = 0.99984, 91.4 – 91.9% CCV Recoveries), QC blanks, duplicate analysis (0%RPD), and blank spike analysis (88.3% – 93.0%). The soluble MS recovery was considerably lower in the reanalysis and still below QC limits, while the post spike and pH-adjusted post spike recoveries were about the same in the re-analysis, as detailed below. There was not a good agreement between the samples and the 1:5 dilution.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the post-digestion spike recovery in QC Sample JC19001-1R soluble MS recovery was lower compared to the initial analyses, as observed below in Table 7, while the insoluble MS recovery, post-digestion and pH-adjusted post spike recoveries were similar to the recoveries in the initial analysis. The insoluble MS recovery in JC19001-1R was still well within the 75-125% QC limits.

Table 7. Hexavalent Chromium Re-analysis MS Recovery Results – JC19001

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97260 X	JC19001-1R	Cr ⁺⁶ , soluble	10.8 %	NJ-	Low
GP97260 X	JC19001-1R	Cr ⁺⁶ , insoluble	84.7 %	----	----
GP97260 X	JC19001-1R	Cr ⁺⁶ , post-digestion spike	69.83 %	NJ-	Low
GP97260 X	JC19001-1R	Cr ⁺⁶ , pH-adjusted post spike	72.34 %	NJ-	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. X – The samples associated with QC Batch GP97260 consist of JC19001-1R through -4R (inclusive).					

Since the soluble MS recovery in QC Batch GP97260 was still below the QC limits (75-125%), the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” for a potential low bias in the ability to recover Cr+6 in this QC batch. The qualified Cr+6 results of the reanalysis are presented below in Table 8 together with the results of the initial Cr+6 results.

Although the soluble MS recovery in QC Batch GP87260 was less than 50%, the associated sample results were qualified as estimated values and flagged with “NJ-“, rather than be rejected, because of several reasons. First, inorganic DV guidelines do not recommend rejection of detected concentrations, even those associated with MS recoveries below 30% (US EPA, 2014). Secondly, the insoluble recovery (84.7%) was within QC limits and may be a better representation of the ability to recover Cr+6 from the soil matrix than that indicated by the soluble MS recovery value, a data usability approach discussed with Mr. Joseph Sanguiliano of the NJDEP. Like the initial Cr+6 analysis, the low soluble MS recovery again suggests a potential low bias in the ability to recover Cr+6 in this QC batch. The fact that the soluble MS recovery in the re-analysis fell from 28 to 10.8%, yet the observed increase in the Cr+6 result in JC19001-3, which increased from 53.9 to 117 mg/kg, is likely attributable to sample non-homogeneity, but also raises the question as to the reliability/credibility of the soluble MS recovery result to appropriately represent the ability to recover Cr+6 from a soil matrix.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC19001-1RT (PPG174_RR_B01R), a QC samples which was analyzed twice with a detected concentration of 0.55 mg/kg and a non-detect result of < 0.47 mg/kg, values well below the SCC of 20 mg/kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. The total organic carbon (TOC) analysis was performed within the 14-day analytical holding time and, hence, the TOC result is not subject to qualification. In accordance with the method, these analyses were performed on the sample experiencing the low spike recoveries. A concentration of total organic carbon (154,000 mg/kg) and the ferrous iron (Fe+2) with a result of 0.73 % were detected in the QC sample JC19001-1RT, thereby indicating the likely presence of a reducing soil matrix in the soil sample, as suggested by the presence of this soil sample below the Eh-pH phase line, as are two of the other three soil samples of this SDG.

The “reducing” conditions in the soil matrix appear supported by the detected TOC concentration and the detected Fe⁺² data in support of the results of the Eh-pH analyses.

Summary for Hexavalent Chromium Analysis – SDGs JC19001

The qualified soil sample results from the initial Cr+6 analysis in SDG JC19001 are presented below in Table 8 alongside those qualified results obtained from the reanalysis of the samples. Both sets of analytical Cr+6 results for samples JC19001-1 through -4 and their reanalysis are still both qualified as estimated values (NJ-) due to a potential low bias, although the soluble MS recovery of the second analysis exhibited a lower recovery in the re-analysis that was performed within the 30-day holding time. The Cr+6 concentrations determined during the re-analysis of samples in SDG JC19001 differ slightly from those of the initial analysis, but, with the exception of Sample JC19001-3 from oxidizing soil conditions, the remaining three samples from a reducing soil matrix are still well below the SCC of 20 mg/kg.

Table 8. Comparison of Qualified Cr+6 Results in JC19001 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC19001 Result (mg/kg)	DV Qualifier	JC19001-R Results (mg/kg)	DV Qualifier
PPG174_RR_B01R	JC19001-1	Cr+6	0.55	NJ-	< 0.47	NJ-
PPG174_RR_SW01R	JC19001-2	Cr+6	5.7	NJ-	2.3	NJ-

Client ID	Laboratory Sample ID	Analyte	JC19001 Result (mg/kg)	DV Qualifier	JC19001-R Results (mg/kg)	DV Qualifier
PPG174 FCCR	JC19001-3	Cr+6	53.9	NJ-	117	NJ-
PPG174 DUP01	JC19001-4	Cr+6	7.5	NJ-	3.9	NJ-
mg/kg - milligrams per kilogram < -The analyte was analyzed for but was not detected above the stated reporting limit. NJ- - The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.						

Although the Cr+6 results of the initial analysis may be subject to rejection because the soluble MS recovery of 28.5% was below the 50% criterion where DV guidelines recommend rejection of associated sample results (NJDEP, 2009), the Cr+6 results in both the initial and re-analysis were qualified as estimated values (NJ-) and not rejected due to a data usability approach and professional judgement based on the following considerations.

First of all, Cr+6 concentrations were detected initially in each of the four post-excavation samples, and in three of the four samples of the re-analysis. Inorganic analysis data validation guidelines do not recommend rejection of detected results (US EPA, 2014). Thus, the only result that might be considered for rejection would be the non-detect Cr+6 result in reanalysis sample JC19001-1R. This sample exhibits a “reducing” soil environment represented by its position below the Eh-pH phase diagram line and the high TOC concentration (154,000 mg/kg) with a 0.73% ferrous iron result. Furthermore, the total chromium concentration is a low value of 21.7 mg/kg, making it highly unlikely that this sample with “reducing” soil conditions and a low chromium concentration would experience an oxidation of chromium that could approach the SCC of 20 mg/kg. Hence, it was judged unnecessary to reject this non-detect value, also in part because it supports the initial low concentration (0.55 mg/kg) detected in the initial analysis.

Because of the low Cr+6 results in three samples under “reducing” soil conditions, and the significantly increased Cr+6 result in the reanalysis for the sample experiencing “oxidizing” conditions (JC19001-3R), the variability between the initial and re-analysis Cr+6 concentrations may be due more to sample non-homogeneity, rather than correlated to the soluble MS recoveries. Both analyses were performed within the 30-day analytical holding time.

Because the three samples with the low Cr+6 results are present in conditions which represent “reducing” soil conditions and have total chromium concentrations less than 300 mg/kg, the Cr+6 concentrations in these three soil samples with Cr+6 concentrations below the SCC of 20 mg/kg are not expected to increase significantly that might approach the SCC level. Thus, it was judged unnecessary to reject any of the Cr+6 results in the samples of SDG JC19001 because the Cr+6 results below the SCC are not expected to significantly increase, and the single sample with “oxidizing” soil conditions has to be addressed in additional project activities due to both results being significantly above the action level SCC.

In consideration of the extensive amount of Cr+6 analyses performed at various PPG sites, it appears that the ability to recover Cr+6 from PPG soil samples is correlated more with the insoluble MS recoveries than results of the soluble MS recoveries. Because the insoluble MS recoveries were both similar and within QC limits in the initial, as well as the re-analysis, this provides additional support for qualifying, and not rejecting, the Cr+6 results of the initial analysis, as well as the re-analysis.

The three soil samples experiencing low Cr+6 results under “reducing” soil conditions exhibit total chromium to Cr+6 ratios ranging 25 to 40, values above the 20:1 ratio generally observed at the many PPG sites. Review of hundreds of soil samples from various PPG sites has shown that samples containing 500 mg/kg might contain a corresponding Cr+6 concentration approaching the SCC criterion, though not exceeding it. Soil samples containing chromium and Cr+6 may be found to typically exhibit a ratio of approximately 20:1, similar to ratios observed at various Hudson County sites by Paustenbach, et al. (1991). This further supports the decision to qualify the Cr+6 results of SDG JC19001, rather than consider rejection of these reported results.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

Paustenbach, D.J., Rinehart, W.E., and P.J. Sheehan, 1991, "The Health Hazards Posed by Chromium-Contaminated Soils in Residential and Industrial Areas: Conclusions of an Expert Panel" in ***Regulatory Toxicology and Pharmacology***, 13, pp 195-222 (1991).

US EPA, CLP, 2014, ***"National Functional Guidelines for Inorganic Superfund Data Review"***, OSWER Publication 9335.0-131, EPA-540-R-13-001, August 2014.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***"National Functional Guidelines for Inorganic Superfund Data Review"***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC19001/JC19001A

1. Were the appropriate sample preservation requirements met?..... Yes No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Sample JC19001-4A was diluted 2× for thallium analysis due to the presence of a high interfering element.

Sample JB19001-3 was diluted 5× and re-analysis Sample JC19001-3R was diluted 10× because the Cr+6 concentration exceeded the calibration range.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The nickel results in Samples JC19001-3A and -4A exceeded the IGWSSL of 48 mg/kg, while the Cr+6 results in JC19001-3 and re-analysis sample JC19001-3R exceeded the SCC of 20 mg/kg.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... Yes No
9. Were rejections noted in the non-conformance summary?..... Yes No
Provide a brief explanation.
Not applicable
10. Were rejected data used?..... Yes No
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes No
12. Were the QC Summary Forms reviewed?..... Yes No
13. Internal Standards acceptable..... Yes No
14. MS/MSD acceptable..... Yes No
15. Calibration summaries acceptable..... Yes No
16. Serial dilutions acceptable..... Yes No
17. Inorganic duplicates acceptable..... Yes No
18. LCS recovery acceptable..... Yes No
19. Other QC acceptable?..... Yes No
Provide a brief explanation, if applicable.

The field duplicate sample results for chromium and nickel in samples JC19001-2A and -4A differed by more than 50%RPD and are qualified (“J”) as estimated values.

Refer to DV report tables 2, 3, 5, 6, and 7 for QC details. Qualified sample results are presented in Tables 4 and 8 of this DV report.



DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC19375/JC19375A
Sample Dates: April 29, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Reviewer: Janis V. Giga. Ph.D., REP5554
Report Date: May 30, 2016

This data validation (DV) report presents the data review and result qualifications for three (3) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, from April 29, 2016, for sample delivery group (SDG) JC19375, as well as JC19375A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC19375A and JC19375 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 3 collected post-excavation soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC19375-1A through JC19375-3A (inclusive)
- Hexavalent chromium ("NJ-") in Samples JC19375-1 through JC19375-3 (inclusive)
- Hexavalent chromium ("NJ-") in reanalysis samples JC19375-1R through JC19375-3R (inclusive)

No other sample results in SDG JC19375A and JC19375 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) concentration in JC19375-1 was above the Soil Cleanup Criterion (SCC) of 20 milligrams per kilogram (mg/Kg) in the respective SDG. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Tables 3 and 6 of this DV report.

Sample Receipt

The three (3) post-excavation soil samples collected April 29, 2016, were received intact and appropriately preserved the same day, April 29, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 4.3 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC19375A and JC19375

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-B01	JC19375-1A	4/29/2016	Soil	Metals
PPG174-MAIN-B02	JC19375-2A	4/29/2016	Soil	Metals
PPG174-MAIN-B03	JC19375-3A	4/29/2016	Soil	Metals
PPG174-MAIN-B01	JC19375-1	4/29/2016	Soil	Cr+6
PPG174-MAIN-B02	JC19375-2	4/29/2016	Soil	Cr+6
PPG174-MAIN-B03	JC19375-3	4/29/2016	Soil	Cr+6

Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids.
Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.

The data package presenting the metals data is numbered JC19375A, while the data package for the hexavalent chromium analyses is numbered JC19375.

Data Review

Data, as presented in the analytical data packages SDG JC19375A and JC19375, was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC19375A

The data validation of the metals analytical data in SDG JC19375A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 3 post-excavation soil samples were analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 3 soil samples of SDG JC19375A, all analyte results were below the respective IGWSSL and SRS limits.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP93444 indicating possible matrix interference and/or sample non-homogeneity. All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130%)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard),

with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

The exceptions included the 150% recovery of thallium in CRID4 at 8:12 in analytical sequence MA39303 on April 30, 2016, and the 146.7% and 163.3% recoveries of antimony in CRID1 at 11:01 and CRID2 at 15:07 of analytical sequence MA39309 on May 2, 2016. These are recoveries above the QC limits of 70-130%. However, no thallium or antimony results were subject to qualification because either no soil sample result was directly associated with the affected CRI standard, as well as there is no potential positive bias in a non-detect result, since neither thallium nor antimony was detected in any of the samples of this SDG.

Thallium experienced 88.5% recoveries in the closing continuing calibration verification (CCV) standards CCV14 and CCV15 in analytical sequence MA39303 on April 30, 2017, values below the QC limits of 90-110%. However, no soil sample thallium results were associated with either of these two CCVs.

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), except thallium in CCB3 (3.0 micrograms per liter [µg/L]) at 19:47 of analytical sequence MA39303 on April 30, 2016, and CCB2 at 11:31 (2.1 µg/L) of analytical sequence MA39309 on May 2, 2016. However, no thallium results were subject to qualification because these CCBs were not directly associated with any soil samples and thallium was not detected in any of the associated samples. Hence, no soil sample results warranted qualification for any associated QC blank contamination in SDG JC19375A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35% Relative Percent Difference [RPD])

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for a non-client QC batch sample JC19035-5, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93444 Ω	JC19035-5	Antimony	68.7 %	71.4 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike						

MSD – Matrix spike duplicate.

NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.

Ω – The samples associated with QC Batch MP93444 consist of JC19375-1A through -3A (inclusive).

The antimony results in these three affected soil samples are flagged with “NJ-” due to a potential low bias. The metals concentrations in the non-client QC sample appear to be similar to those typically observed in PPG samples and, therefore, qualification of the associated antimony results was judged appropriate in this case. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 0.5 – 16.7%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated acceptable analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 87.0% - 98.5% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit ≤ 10 %D)

The serial dilution results associated with the soil samples ranged from 0 – 1.6 percent difference (%D), values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC19375A were found to be compliant with the analytical methods for the analysis of metals in the 3 soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP93444 associated with the 3 soil samples: JC19375-1A through JC19375-3A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JC19375A

Sample ID	Lab ID	Analyte	Result (mg/Kg)	DV Qualifier
PPG174-MAIN-B01	JC19375-1A	Antimony	< 2.3	NJ-
PPG174-MAIN-B02	JC19375-2A	Antimony	< 2.3	NJ-

Sample ID	Lab ID	Analyte	Result (mg/Kg)	DV Qualifier
PPG174-MAIN-B03	JC19375-3A	Antimony	< 2.3	NJ-

Key:
mg/Kg - milligrams per kilogram
< -The analyte was analyzed for but was not detected above the stated reporting limit.
NJ- - The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC19375

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the three post-excavation soil samples. The soil samples were re-analyzed in a second QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Data qualifiers

Hexavalent chromium was detected in two of the three soil samples analyzed in SDG JC19375, with the sample Cr+6 result of 68.2 mg/Kg being the highest concentration in the initial analysis and the only result exceeding the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/Kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike, post spike and pH adjusted post spike recoveries in QC Batch GP97338 were outside control limits, as well as in reanalysis QC Batch GP97362. The RPD value for the duplicate analysis in the re-analysis QC Batch GP97362 was above control limits, but the RPD was acceptable due to low duplicate and sample concentrations. There was a good agreement between the sample and 1:5 dilution in the initial, but not the re-analysis. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% CCV Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99982 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 94.7 to 95.9% for the QC batch associated with the analysis of 3 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/Kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery were below the QC limits of 75-125% for QC Batch GP97338 associated with the 3 soil samples of this SDG, as presented below in Table 4. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP97338 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices, while the insoluble MS recovery was well within the QC limits. The post spike and pH-adjusted were also below the QC limits of 85-115%.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC19375

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97338 ¥	JC19375-3	Cr ⁺⁶ , soluble	1.6 %	NJ-	Low
GP97338 ¥	JC19375-3	Cr ⁺⁶ , insoluble	94.0 %	----	----
GP97338 ¥	JC19375-3	Cr ⁺⁶ , post-digestion spike	36.07 %	NJ-	Low
GP97338 ¥	JC19375-3	Cr ⁺⁶ , pH-adjusted post spike	27.76 %	NJ-	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. ¥ – The samples associated with QC Batch GP97338 consist of JC19375-1 through -3 (inclusive).					

The Cr+6 results qualified for low spike recoveries are flagged with “NJ-” (NJDEP, 2009; US EPA, 2012), as tabulated below in Table 6, together with the qualified results from the re-analysis of this QC batch. Qualification of these Cr+6 results is discussed in the Summary section at the end of the Cr+6 review.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JC19375-3 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-CCC02) was 0%RPD, a value below the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (2.0%RPD) and pH (2.4%RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 98.0% and 106.3% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as serial dilution is not a requirement of the analytical method and serial dilution is not addressed in DV guidelines (NJDEP,

2009). A 1:5 dilution was actually performed and there was good agreement between the sample and the 1:5 diluted aliquot. No additional qualification is warranted.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/Kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)
 C = Wet weight of sample (Kg)
 D = % Solids/100
 E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-MAIN-B01 (JC19375-1) was listed as 68.2 mg/Kg on the reporting form and 0.3143 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/Kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/Kg)} = \frac{0.3143 \text{ mg/L} \times 0.1 \text{ L} \times 5}{0.00257 \text{ Kg} \times 89.7/100} = \frac{0.1572}{0.0023053} = 68.1693 \text{ mg/Kg}$$

$$\text{Cr}^{+6} \text{ (mg/Kg)} = 68.2 \text{ mg/Kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 68.2 mg/Kg for Sample PPG174-MAIN-B01 was correctly reported. This was the highest detected Cr+6 concentration of the two detected results for the 3 soil samples of this SDG, a value above the SCC of 20 mg/Kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Each of the three soil samples were observed to fall above the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of an “oxidizing” soil environment. The Cr+6 sample concentrations in an oxidizing soil may increase in value, provided the conditions are suitable for oxidation and provided there is a significant concentration of chromium available. The total chromium concentration in JC19375-3 associated with a non-detect Cr+6 result was only 20.1 mg/Kg, thereby making it less likely that the Cr+6 concentration would increase to any significant degree that could approach the SCC of 20 mg/Kg. Review of many PPG soil samples experiencing “oxidizing” soil samples tend to exhibit, with few exceptions, total chromium to Cr+6

ratios (Cr:Cr+6) ranging from approximately 7 to 75, thereby supporting the likelihood that the non-detect Cr+6 result in JC19375-3 will not oxidize to a concentration approaching the SCC of 20 mg/Kg.

Summary for Hexavalent Chromium Analysis – SDG JC19375

Since the soluble MS spike recovery of 1.6% was below QC limits in the QC sample of QC Batch GP97338, the soil sample in this QC batch required reanalysis. Therefore, the Cr+6 results for the associated samples were qualified following the DV review and flagged with “NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix. Consequently, the soil samples of this QC Batch GP97338 were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analysis in JC19375.”

Cr+6 Re-analyses in SDG JC19375

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the batch consisting of 3 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC19375-1R through -3R in QC Batch GP97362, including the calibrations (r = 0.99975, 92.8 – 93.6% CCV Recoveries), QC blanks, duplicate analysis (≤ 2 × CRQL), and blank spike analysis (87.3 and 84.5%). The soluble MS recovery was considerably improved in the reanalysis, while the post spike and pH-adjusted post spike recoveries were improved as well, as detailed below.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the soluble MS recovery in QC Sample JC19375-3R was considerably better than the initial analysis, but still under the QC limits, while the insoluble MS recovery was lower than the initial recovery, but still within the QC limits of 75-125%, as observed below in Table 5. The post-digestion spike and pH-adjusted post spike recoveries improved significantly in the re-analysis.

Table 5. Hexavalent Chromium Re-analysis MS Recovery Results – JC19375

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97362 X	JC19375-3R	Cr ⁺⁶ , soluble	23.7 %	NJ-	Low
GP97362 X	JC19375-3R	Cr ⁺⁶ , insoluble	81.2 %	----	----
GP97362 X	JC19375-3R	Cr ⁺⁶ , post-digestion spike	66.79 %	NJ-	Low
GP97362 X	JC19375-3R	Cr ⁺⁶ , pH-adjusted post spike	60.93 %	NJ-	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. X – The samples associated with QC Batch GP97362 consist of JC19375-1R through -3R (inclusive).					

Since the soluble MS recovery in QC Batch GP97362 was still below the QC limits (75-125%), the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” for a potential low bias in the ability to recover Cr+6 in this QC batch. The qualified Cr+6 results of the reanalysis are presented below in Table 6 together with the results of the initial Cr+6 results.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots. The difference between the duplicate soil sample aliquot concentrations for Cr+6 in the sample aliquots was listed as 200%RPD. Although this RPD value exceeded 35%, the analytical precision results were acceptable because of the low sample concentrations where the difference (0.54 mg/Kg) between the raw concentration values was less than twice the reporting limit (2 × 0.47 mg/Kg). Hence, the Cr+6 results in the associated samples were not qualified for the duplicate analysis result and analytical precision is considered acceptable in the re-analysis.

Summary for Hexavalent Chromium Analysis – SDGs JC19375

The qualified soil sample results from the initial Cr+6 analysis in SDG JC19375 are presented below in Table 6 alongside those qualified results obtained from the re-analysis of the samples. Both sets of analytical Cr+6 results for samples JC19375-1 through -3 and their re-analysis are still both qualified as estimated values (NJ-) due to a potential low bias, as the soluble MS recoveries were both below QC limits. The second analysis exhibited improved MS recoveries, except the insoluble MS recovery, which was slightly lower, but still within QC limits.

Table 6. Comparison of Qualified Cr+6 Results in JC19375 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC21391 Result (mg/Kg)	DV Qualifier	JC21391-R Results (mg/Kg)	DV Qualifier
PPG174-MAIN-B01	JC19375-1	Cr+6	68.2	NJ-	17.7	NJ-
PPG174-MAIN-B02	JC19375-2	Cr+6	0.61	NJ-	1.8	NJ-
PPG174-MAIN-B03	JC19375-3	Cr+6	< 0.47	NJ-	< 0.47	NJ-

mg/Kg - milligrams per kilogram
< –The analyte was analyzed for but was not detected above the stated reporting limit.
NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

Professional judgement was applied in not rejecting the initial analysis result for JC19375-1 (68.2 mg/Kg) because the Cr+6 result was considerably above the SCC of 20 mg/Kg and inorganic DV guidelines do not recommend rejection of detected concentrations (US EPA, 2014). Regardless of how low the soluble MS recovery appears, the detected result is significant and warrants additional attention. The re-analysis result for JC19375-1R was also not rejected, because the result was also detected, though lower and below the SCC of 20 mg/Kg despite the improved soluble MS recovery, and because the elevated concentration in the initial analysis warrants additional project activity concerning this sampling location.

The initial and re-analysis results for JC19375-2 and -2R were also not rejected because they also are detected concentrations (US EPA, 2014). The total chromium concentration for this sampling location was 130 mg/Kg, a value below 500 mg/Kg, a guideline value below which PPG samples generally do not exhibit corresponding Cr+6 concentrations above the SCC of 20 mg/Kg.

Review of hundreds of soil samples from PPG sites has shown that samples containing less than 500 mg/Kg total chromium rarely contain a corresponding Cr+6 concentration approaching the SCC criterion. Soil samples containing chromium and Cr+6 may be found to typically exhibit a ratio of approximately 20:1, similar to ratios observed at various Hudson County sites by Paustenbach, et al. (1991). The present ratio of total chromium to Cr+6 for Sample JC19375-2 is greater than 200 which decreased to 72 for the re-analysis. The fact that the Cr+6 concentration slightly increased (3-fold) upon reanalysis with the 15-fold improvement in the soluble MS recovery

seems to confirm the presence of a low Cr+6 concentration in JC19375-1, further supporting the decision to qualify, not reject, either of the detected Cr+6 results for sampling location JC19375-2.

Professional judgement based on data usability concepts was applied in not rejecting the two non-detect results from location JC19375-3, because the corresponding total chromium concentration was only 20.1 mg/Kg. In PPG samples observed to exhibit "oxidizing" soil conditions rarely exhibit Cr:Cr+6 ratios below 7. Hence, even if the chromium were to experience significant oxidation, it is unlikely that a Cr+6 concentration would exceed the SCC. Furthermore, the non-detect Cr+6 result of the initial analysis was confirmed in the reanalysis, despite a significant increase in the soluble MS recovery, as well as those increases of the post spike and pH-adjusted spike recoveries in the re-analysis. Additionally, the insoluble MS recoveries were both within QC limits, and experience at PPG sites suggests that the insoluble MS recovery may be a better indicator of the ability to recover Cr+6 from the soil matrix than the soluble MS recovery result.

Hence, the reported sample results are usable within the context of the applied qualifications, based on these described data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

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New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

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New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

Paustenbach, D.J., Rinehart, W.E., and P.J. Sheehan, 1991, "The Health Hazards Posed by Chromium-Contaminated Soils in Residential and Industrial Areas: Conclusions of an Expert Panel" in ***Regulatory Toxicology and Pharmacology***, 13, pp 195-222 (1991).

US EPA, CLP, 2014, ***"National Functional Guidelines for Inorganic Superfund Data Review"***, OSWER Publication 9335.0-131, EPA-540-R-13-001, August 2014.

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US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC19375/JC19375A

- 1. Were the appropriate sample preservation requirements met?..... Yes No
- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.
- 3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Sample JC19375-1 was diluted 5-fold because the detected Cr+6 concentration was above the calibration range.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.
- 5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The Cr+6 result in JC19375-1 exceeded the SCC of 20 mg/Kg.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.
- 7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2, 4, and 5 for QC details. Qualified sample results are presented in Tables 3 and 6 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC19696/JC19696A
Sample Dates: May 5, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga. Ph.D., REP5554
Report Date: June 21, 2016

This data validation (DV) report presents the data review and result qualifications for six (6) soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on May 5, 2016, for sample delivery group (SDG) JC19696, as well as JC19696A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC19696A and JC19696 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 6 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC19696-1A through JC19696-6A (inclusive)
- Hexavalent chromium ("NJ-") in Samples JC19696-1 through JC19696-6 (inclusive)
- Hexavalent chromium ("NJ-") in reanalysis samples JC19696-1R through JC19696-6R (inclusive)

No other sample results in SDG JC19696A and JC19696 required qualification, based on the acceptable remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) concentrations were below the Soil Cleanup Criterion (SCC) in the respective SDGs, except JC19696-4 and re-analysis sample JC19696-4R. A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 3 and Table 6 of this DV report.

Sample Receipt

The six (6) soil samples collected May 5, 2016, were received intact and appropriately preserved May 5, 2016, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 5.2 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC19696A and JC19696

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-B01R	JC19696-1A	5/5/16	Soil	Metals
PPG174-MAIN-B04	JC19696-2A	5/5/16	Soil	Metals
PPG174-MAIN-B05	JC19696-3A	5/5/16	Soil	Metals
PPG174-MAIN-B06	JC19696-4A	5/5/16	Soil	Metals
PPG174-MAIN-B07	JC19696-5A	5/5/16	Soil	Metals
PPG174-MAIN-B08	JC19696-6A	5/5/16	Soil	Metals
PPG174-MAIN-B01R	JC19696-1	5/5/16	Soil	Cr+6
PPG174-MAIN-B01R	JC19696-1RT	5/5/16	Soil	TOC, SS, Fe2+
PPG174-MAIN-B04	JC19696-2	5/5/16	Soil	Cr+6
PPG174-MAIN-B05	JC19696-3	5/5/16	Soil	Cr+6
PPG174-MAIN-B06	JC19696-4	5/5/16	Soil	Cr+6
PPG174-MAIN-B07	JC19696-5	5/5/16	Soil	Cr+6
PPG174-MAIN-B08	JC19696-6	5/5/16	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting the metals data is numbered JC19696A, while the data package for the hexavalent chromium analyses is numbered JC19696. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC19696 together with the supplemental total organic carbon (TOC), sulfide screen and ferrous iron. The samples data were validated for the five target metals (antimony, chromium, nickel, thallium, and vanadium), as were the hexavalent chromium data, and supplemental TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC19696A and JC19696 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9335.0-131, EPA540-R-013-001, August 2014 (US EPA, 2014).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).

- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC19696A

The data validation of the metals analytical data in SDG JC19696A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 6 soil samples were analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples, and are covered by this data validation. Of the sample metals results detected in the 6 samples of SDG JC19696A, no result exhibited a concentration above the IGWSSL or SRS, whichever was more stringent.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC Batch MP93556 indicating possible matrix interference. The case narrative identified the serial dilution result being outside QC limits for antimony and thallium; however, the percent difference (%D) results were acceptable due to low initial sample concentrations (< 50 times instrument detection limit [IDL]). The case narrative also stated that the detection limit for vanadium in JC19696-4A was elevated. However, the detection limit for chromium in this sample was also elevated, but both chromium and vanadium were detected in this sample. All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Consequently, no soil sample or field blank results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs) in most analytical sequences, except that thallium was detected in CCB5 at 15:02 at a concentration of 2.3 micrograms per liter (µg/L) in analytical sequence MA39352. However, the detection of thallium in the CCB5 did not affect the soil sample results, since thallium was not detected in the associated samples and QC samples. Hence, no soil sample results warranted qualification for any associated QC blank contamination in SDG JC19696A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35% Relative Percent Difference [RPD])

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony of this SDG are outside the QC limits of 75 - 125% for the QC sample JC19696-3A as summarized below in Table 2. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample results subject to qualification were flagged with “N” to indicate that the result is associated with QC recovery outside QC limits and the associated antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93556 Ω	JC19696-3A	Antimony	57.5 %	55.3 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias. Ω – The samples associated with QC Batch MP93556 consist of JC19696-1A through -6A (inclusive).						

The antimony results in the affected soil samples are flagged with “NJ-” due to a potential low bias, and the qualified results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 2.1 – 3.4%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 93.6% - 97.0% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative identified the serial dilution result being outside QC limits for antimony and thallium; however, the percent difference (%D) results were acceptable due to low initial sample concentrations (< 50 times IDL). The serial dilution results associated with the soil samples ranged from 0.1 – 5.0%D for the other three analytes, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

The only sample analysis that required dilution included chromium and vanadium analysis in JC19696-4A where a 2× dilution was performed due to the presence of an interfering element, such that the reporting limits for chromium and vanadium were raised. However, sample results were not affected since these analytes were detected in this sample at concentrations below the respective IGWSSL and SRS. Thus, all reporting limits were below the respective SRS and IGWSSL values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC19696A were found to be compliant with the analytical methods for the analysis of metals in the 6 soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP93556 associated with the 6 soil samples. Therefore, the antimony results are qualified as estimated values (flagged "NJ-") in the associated soil samples due to a potential low bias, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JC19696A

Sample ID	Lab ID	Analyte	Result (mg/Kg)	DV Qualifier
PPG174-MAIN-B01R	JC19696-1A	Antimony	< 2.2	NJ-
PPG174-MAIN-B04	JC19696-2A	Antimony	< 2.4	NJ-
PPG174-MAIN-B05	JC19696-3A	Antimony	< 2.3	NJ-
PPG174-MAIN-B06	JC19696-4A	Antimony	5.9	NJ-
PPG174-MAIN-B07	JC19696-5A	Antimony	< 2.3	NJ-
PPG174-MAIN-B08	JC19696-6A	Antimony	< 2.2	NJ-

Key:
 mg/kg - milligrams per kilogram
 < -The analyte was analyzed for but was not detected above the stated reporting limit.
 NJ- - The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC19696

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the six soil samples. The soil samples were reanalyzed for Cr+6 in another QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Quantitation checks
- √ Data qualifiers
- Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Calibration verification
- √ Data package completeness

Hexavalent chromium was detected in each of the six soil samples analyzed in SDG JC19696 and six of the six re-analyzed samples, with all sample Cr+6 results less than or equal to 4.7 mg/Kg, values well below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/Kg, except for the 167 mg/Kg result in JC19696-4 and 176 mg/Kg in its re-analysis.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike, post spike and pH adjusted post spike

recoveries in QC Batch GP97439 were outside control limits, as well as in reanalysis QC Batch GP97678. The RPD value for the duplicate analysis in the analysis QC Batch GP97439 was above control limits; however, the RPD value was acceptable due to low sample and duplicate concentrations. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99995 for the soil samples analysis, and 0.99984 for the re-analysis, values greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 96.0% to 96.5% for the QC batch associated with the initial analysis of 6 soil samples, and 93.5 to 94.4% for the reanalysis, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/Kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery were below the QC limits of 75-125% for QC Batch GP97439 associated with the 6 soil samples of this SDG, while the post-digestion spike and pH-adjusted post spike recoveries were below the 85-115% QC limits, as presented below in Table 4. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP97439 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. The remaining insoluble MS recovery was within QC limits.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC19696

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97439 ¥	JC19696-1	Cr ⁺⁶ , soluble	42.5 %	NJ-	Low
GP97439 ¥	JC19696-1	Cr ⁺⁶ , insoluble	90.7 %	----	----
GP97439 ¥	JC19696-1	Cr ⁺⁶ , post-digestion spike	70.8 %	NJ-	Low
GP97439 ¥	JC19696-1	Cr ⁺⁶ , pH-adjusted post spike	68.9 %	NJ-	Low

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
 MS – Matrix spike
 Cr⁺⁶ – Hexavalent chromium
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
 ¥ – The samples associated with QC Batch GP97439 consist of JC19696-1 through -6 (inclusive).

The Cr+6 results qualified for low spike recoveries are flagged with “NJ-”, as tabulated below in Table 6, together with the qualified results from the re-analysis of this QC batch.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JC19696-1. The difference between the duplicate soil sample aliquots for Cr+6 in soil this sample (PPG174-MAIN-B01R) was 39.5%RPD, a value above the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (US EPA, 2010; AECOM, 2010). However, the result was acceptable because the difference was less than the reporting limit of 0.47 mg/Kg due to the low sample concentration and its duplicate result which meets the QC limit of being less than twice the reporting limit for soil samples (US EPA, 2010) such that the associated results are not subject to

qualification. The %RPD values for redox potential (7.0%RPD) and pH (1.3%RPD) displayed acceptable analytical precision results. Because the difference in the duplicate analysis for Cr+6 was below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 82.8% and 81.3% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as serial dilution is not a requirement of the analytical method and serial dilution is not addressed in DV guidelines (NJDEP, 2009). A 1:5 dilution was actually performed on Sample JC19696-1 and there was poor agreement between the sample and the diluted aliquot. However, even though the results may be considered for qualification, the Cr+6 results are already qualified as estimated values and are flagged with "NJ-" for the low spike recoveries. Additional qualification is not warranted in an effort to avoid redundancy of qualifiers.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/Kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (Kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-MAIN-B06 (JC19696-4) was listed as 167 mg/Kg on the reporting form and 0.3322 mg/L on the quantitation report in the raw data for a 10-fold dilution. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/Kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/Kg)} = \frac{0.3322 \text{ mg/L} \times 0.1 \text{ L} \times 10}{0.00250 \text{ Kg} \times 79.4/100} = \frac{0.3322}{0.0019850} = 167.3552 \text{ mg/Kg}$$

$$\text{Cr}^{+6} \text{ (mg/Kg)} = 167 \text{ mg/Kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 167 mg/Kg for Sample PPG174-MAIN-B06 was correctly reported. This was the highest

detected Cr+6 concentration of the six detected results for the 6 soil samples of this SDG, a value considerably above the SCC of 20 mg/Kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

All six soil samples were observed to fall below or near the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentrations are also not expected to increase to levels approaching the SCC of 20 mg/Kg, because the total chromium concentrations are, with the exception of the 1800 mg/Kg in JC19696-4, all less than 38 mg/Kg, thereby making it highly unlikely that Cr+6 concentrations would increase to any significant degree. The Cr+6 result in JC19696-4 (167 mg/Kg) is considerably above the SCC and will need to be addressed in the remediation phase of the project.

Hence, based on the sample total chromium and Cr+6 concentrations, it is highly unlikely that any of the affected samples in the “reducing” zone would approach the SCC for Cr+6 of 20 mg/Kg due to limitation created by the low total chromium concentrations available for potential oxidation, except for the elevated Cr+6 result in JC19696-4.

Summary for Hexavalent Chromium Analysis – SDG JC19696

Since the soluble MS spike recovery of 42.5% was below QC limits in the QC samples of QC Batch GP97439, as well as below 50%, the soil samples in this QC batch required reanalysis. The remaining QC results associated with the hexavalent chromium analysis were within QC limits, except for the low post spike and pH-adjusted post spike recoveries. Therefore, the Cr+6 results for the six samples of this QC batch in SDG JC19696 were qualified following the DV review and flagged with “NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix. Consequently, the soil samples of this QC batch were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analyses in SDG JC19696.”

Cr+6 Re-analyses in SDG JC19696

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the batch consisting of 6 soil samples are summarized below. The QC requirements were met during the reanalysis of samples JC19696-1R through -6R in QC Batch GP97678, including the calibration ($r = 0.99984$; and 93.5 – 94.4% CCV Recoveries), QC blanks, duplicate analysis (< 12 %RPD), and blank spike analysis (84.9% – 94.3%). The soluble MS recovery was considerably lower in the reanalysis, as was the insoluble MS recovery, while the post spike and pH-adjusted post spikes were similar to the initial analysis, as detailed below.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the soluble and insoluble MS recoveries in QC Sample JC19696-1R were lower compared to the initial analyses, particularly in the soluble spike, as observed below in Table 5. The insoluble MS recovery in JC19696-1R was still within the 75-125% QC limits.

Table 5. Hexavalent Chromium Re-analysis MS Recovery Results – JC19696

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97678 X	JC19696-1R	Cr ⁺⁶ , soluble	2.8 %	NJ-	Low
GP97678 X	JC19696-1R	Cr ⁺⁶ , insoluble	76.9 %	----	----
GP97678 X	JC19696-1R	Cr ⁺⁶ , post-digestion spike	72.2 %	NJ-	Low
GP97678 X	JC19696-1R	Cr ⁺⁶ , pH-adjusted post spike	71.8 %	NJ-	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium X – The samples associated with QC Batch GP97678 consist of JC19696-1R through -6R (inclusive).					

Since the soluble MS recovery in QC Batch GP97678 was still below the QC limits (75-125%), as well as below 50%, the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” for a potential low bias in the ability to recover Cr+6 in this QC batch. The qualified Cr+6 results of the reanalysis are presented below in Table 6 together with the results of the initial Cr+6 results. Qualification of Cr+6 results is explained below in the Summary section.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots. The difference between the duplicate soil sample aliquot concentrations for Cr+6 in the sample aliquots was an acceptable 11.1%RPD. Hence, the Cr+6 results in the associated samples were not qualified for the duplicate analysis result and analytical precision is considered acceptable in the re-analysis.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC19696-1RT (PPG174-MAIN-B01R), a QC sample which was analyzed twice with detected Cr+6 concentrations of 1.0 and 1.9 mg/Kg for the analyses, values well below the SCC of 20 mg/Kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. The total organic carbon (TOC) analysis of JC19696-1RT was performed within the 14-day analytical holding time, but the TOC analysis was analyzed using multiple injections (4 rather than 2), which may indicate sample non-homogeneity. However, professional judgement was applied in not qualifying the TOC result because the result was used as supplemental information on the redox condition of the soil matrix and the results of the multiple injections were reasonably comparable with the average result being of significant magnitude (623,000 mg/Kg).

In accordance with the analytical method, these analyses were performed on the sample experiencing the low spike recoveries. A concentration of total organic carbon (623,000 mg/Kg) was detected in the QC sample in JC19696-1RT, thereby indicating the likely presence of a

reducing soil matrix in the soil sample, as suggested by the presence of this soil sample below the Eh-pH phase line, as are the other five soil samples of this SDG. Although the ferrous iron (Fe+2) result was a non-detect result of < 0.2 %, the presence of a considerable organic matter content and the samples appearing below the Eh-pH phase line demonstrate a “reducing” soil matrix.

The “reducing” conditions in the soil matrix appear demonstrated by the high detected TOC concentration in support of the results of the Eh-pH analyses.

Summary for Hexavalent Chromium Analysis – SDGs JC19696

The qualified soil sample results from the initial Cr+6 analysis in SDG JC19696 are presented below in Table 6 alongside those qualified results obtained from the reanalysis of the samples. Both sets of analytical Cr+6 results for samples JC19696-1 through -6 and their reanalysis are still both qualified as estimated values (NJ-) due to a potential low bias, although the soluble MS recovery of the second analysis exhibited a considerably lower recovery in the re-analysis that was performed within the 30-day holding time.

Note that even though the soluble MS recovery decreased from 42.5% in the initial analysis to only 2.8% in the re-analysis, the resultant Cr+6 concentrations in the re-analysis were slightly higher than in the initial analysis in five of the six samples. Hence, this tends to suggest that the ability to recover Cr+6 from the sample matrix is not closely tied to the soluble MS recovery result. The Cr+6 concentrations determined during the re-analysis of samples in SDG JC19696 differ slightly from those of the initial analysis, but are reasonably consistent and all, except for JC19696-4, are still well below the SCC of 20 mg/Kg.

Table 6. Comparison of Qualified Cr⁺⁶ Results in JC19696 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC19696 Result (mg/Kg)	DV Qualifier	JC19696-R Results (mg/Kg)	DV Qualifier
PPG174-MAIN-B01R	JC19696-1	Cr+6	1.0	NJ-	1.9	NJ-
PPG174-MAIN-B04	JC19696-2	Cr+6	2.4	NJ-	3.5	NJ-
PPG174-MAIN-B05	JC19696-3	Cr+6	0.83	NJ-	4.7	NJ-
PPG174-MAIN-B06	JC19696-4	Cr+6	167	NJ-	176	NJ-
PPG174-MAIN-B07	JC19696-5	Cr+6	2.3	NJ-	1.9	NJ-
PPG174-MAIN-B08	JC19696-6	Cr+6	1.3	NJ-	2.8	NJ-
mg/Kg – milligrams per kilogram NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.						

Even though the soluble MS recoveries in both the initial and the re-analysis were both less than 50%, sample results were not rejected, but rather qualified as estimated values based on the following data usability considerations. The Cr+6 results were all detected concentrations in each of the samples, and with few exceptions, DV guidelines do not recommend rejection of detected sample results (US EPA, 2014). The insoluble MS recoveries were within QC limits and may be a better representation of the ability of the method to recover Cr+6 from the soil matrix, especially one that exhibits “reducing” soil conditions which do not favor oxidation of chromium to Cr+6. Additionally, the total chromium concentrations in each of the samples, except JC19696-4, were less than 38 mg/Kg, making it highly unlikely that the SCC of 20 mg/Kg would be approached for these samples under a “reducing” environment. Additionally, despite the considerable decrease in the soluble MS recovery in the re-analysis, the detected Cr+6 results in the re-analysis were quite

similar to those of the initial analysis, seemingly placing less importance of the soluble MS recovery results on the interpretation/qualification of the reported Cr+6 results.

The total chromium concentration in Sample JC19696-4 was 1,800 mg/Kg, which likely contributed to the elevated detected Cr+6 concentration of 167 mg/Kg, despite the reducing soil conditions. A general “rule of thumb” for the ratio of total chromium to Cr+6 under reducing soil conditions has been observed in PPG samples to be in the range of 10:1 to 20:1 for total chromium:Cr+6 ratios.

This observation is not unlike results posted in an article entitled “The Health Hazards Posed by Chromium-Contaminated Soils in Residential and Industrial Areas: Conclusions of an Expert Panel” in *Regulatory Toxicology and Pharmacology*, 13, pp 195-222 (Paustenbach, et al.,1991), presenting results of a remedial investigation performed by Environmental Science and Engineering (1989). Soils from 42 sites consisting of residential and industrial sites in Hudson County were analyzed focusing on those considered worst-case sites, likely containing slag fill material. The average Cr+6 soil concentration at these sites was 2.6% of the average total chromium concentration, such that if a soil contained 1000 parts per million (ppm) of total chromium, then approximately 26 ppm was in the form of Cr+6. The highest chromium concentrations were found to be contained at or near the surface.

These findings are not inconsistent with the observations made from review of the many PPG samples collected at Sites 63/65 and 174. Hence, if the Cr+6 concentration were to approach the SCC of 20 mg/Kg, then the soil may be expected to need to contain approximately 500 mg/Kg total chromium, based on this generalized ratio. Review of PPG Cr+6 data covering various sites suggests that soils with less than 500 mg/Kg total chromium are not expected to contain corresponding Cr+6 concentrations that would approach the SCC of 20 mg/Kg. Since the soils of SDG JC19696, except sample JC19696-4, contain less than 38 mg/kg, the soils of this SDG are not expected to approach the SCC of 20 mg/kg, supporting the decision to qualify the Cr+6 results of JC19696, and not reject them despite the low recoveries of the soluble MS being less than 50%.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

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ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC19696/JC19696A

- 1. Were the appropriate sample preservation requirements met?..... Yes No
- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.
- 3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Samples JC19696-4A was diluted 2× for chromium and vanadium analysis due to the presence of a high interfering element. Sample JC19696-4 and the reanalysis (JC19696-4R) were diluted 10× because the Cr+6 concentrations exceeded the calibration range.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.
- 5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The Cr+6 results in Sample JC19696-4 and the re-analysis sample JC19696 -4R exceeded the SCC of 20 mg/Kg.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.
- 7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.
- Not applicable**
10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2, 4, and 5 for QC details. Qualified sample results are presented in Tables 3 and 6 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC19782/JC19782A
Sample Dates: May 6, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Reviewer: Janis V. Giga. Ph.D., REP5554
Report Date: May 20, 2016

This data validation (DV) report presents the data review and result qualifications for seven (7) soil samples and one (1) field blank (FB) collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on May 6, 2016, for sample delivery group (SDG) JC19782, as well as JC19782A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC19782A and JC19782 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 7 collected soil samples and one field blank.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC19782-1A through JC19782-7A (inclusive)

No other sample results in SDG JC19782A and JC19782 required qualification, based on the acceptable remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below. No hexavalent chromium results for the 7 soil samples and 1 field blank of SDG JC19782 were qualified following the DV review, because all QC results were within method QC limits.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except the nickel and vanadium results in two samples (JC19782-1A and 19782-3A), while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Table 3 of this DV report.

Sample Receipt

The seven (7) soil samples and one (1) field blank collected May 6, 2016, were received intact and appropriately preserved the same day, May 6, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 5.3 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC19782A and JC19782

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-SW01	JC19782-1A	5/6/2016	Soil	Metals
PPG174-MAIN-SW02	JC19782-2A	5/6/2016	Soil	Metals
PPG174-MAIN-SW03	JC19782-3A	5/6/2016	Soil	Metals
PPG174-MAIN-SW04	JC19782-4A	5/6/2016	Soil	Metals
PPG174-MAIN-SW05	JC19782-5A	5/6/2016	Soil	Metals
PPG174-CCC01	JC19782-6A	5/6/2016	Soil	Metals
PPG174-CCC02	JC19782-7A	5/6/2016	Soil	Metals
PPG174-FB02	JC19782-8A	5/6/2016	Aqueous	Metals
PPG174-MAIN-SW01	JC19782-1	5/6/2016	Soil	Cr+6
PPG174-MAIN-SW02	JC19782-2	5/6/2016	Soil	Cr+6
PPG174-MAIN-SW03	JC19782-3	5/6/2016	Soil	Cr+6
PPG174-MAIN-SW04	JC19782-4	5/6/2016	Soil	Cr+6
PPG174-MAIN-SW05	JC19782-5	5/6/2016	Soil	Cr+6
PPG174-CCC01	JC19782-6	5/6/2016	Soil	Cr+6
PPG174-CCC02	JC19782-7	5/6/2016	Soil	Cr+6
PPG174-FB02	JC19782-8	5/6/2016	Aqueous	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JC19782A, while the data package for the hexavalent chromium analyses is numbered JC19782.

Data Review

Data, as presented in the analytical data packages SDG JC19782A and JC19782 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) *“National Functional Guidelines for Inorganic Data Review”*, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA *“ICP-AES Data Validation, SOP No. HW-2a, Revision 15”* (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.

- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC19782A

The data validation of the metals analytical data in SDG JC19782A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 7 soil samples and 1 field blank were analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 7 samples of SDG JC19782A, the nickel and vanadium results in two samples (JC19782-1A and JC19782-3A) exhibited a concentration above the IGWSSL of 48 milligrams per kilogram (mg/kg) for nickel and the SRS of 78 mg/kg for vanadium.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP93576 indicating

possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution result for chromium was outside control limits in QC Batch MP93483 associated with the field blank; however, the percent difference (%D) result was acceptable due to a low initial sample chromium concentration (< 50 times instrument detection limit [IDL]). All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil samples and field blank analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

The exception consisted of the 0% recovery of antimony in CRID1 at 10:21 in analytical sequence MA39366 associated with only the QC samples associated with the field blank. However, this affected contract required detection limit (CRDL) standard was not associated with any soil samples or the field blank of this SDG. These QC samples associated with the field blank were analyzed between acceptable CDRL standards. Thus, no sample results required qualifications for calibration issues.

Consequently, no soil sample or field blank results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks, the continuing calibration blanks (CCBs) or the field blank at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC19782A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for PPG QC batch sample JC19782-7A, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits, including those of QC Batch MP93483 (aqueous matrix).

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93576 Ω	JC19782-7A	Antimony	52.8 %	53.7 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ω – The samples associated with QC Batch MP93576 consist of JC19782-1A through -7A (inclusive).						

The antimony results in the seven affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate soil sample aliquots and one pair of aqueous sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 4.5 – 5.7%RPD for soil samples and 0.5 – 5.2 %RPD for the batch QC sample associated with the field blank analysis with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 95.5% - 100.5% for the soil sample metals analysis, and 91.5 – 102.0% for the aqueous matrix.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative stated that the RPD serial dilution result for chromium was outside control limits in QC Batch MP93483 associated with the field blank; however, the percent difference (%D) result was acceptable due to a low initial sample chromium concentration (< 50 times IDL). The serial dilution results associated with the soil samples ranged from 0 – 5.3%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). Hence, no sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limits.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC19782A were found to be compliant with the analytical methods for the analysis of metals in the seven soil samples and one field blank using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP93576 associated with the 7 soil samples: JC19782-1A through JC19782-7A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias in the ability to recover antimony from the soil sample matrix, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JC19782A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-SW01	JC19782-1A	Antimony	< 2.5	NJ-
PPG174-MAIN-SW02	JC19782-2A	Antimony	< 2.4	NJ-
PPG174-MAIN-SW03	JC19782-3A	Antimony	< 2.3	NJ-
PPG174-MAIN-SW04	JC19782-4A	Antimony	< 2.2	NJ-
PPG174-MAIN-SW05	JC19782-5A	Antimony	< 2.4	NJ-
PPG174-CCC01	JC19782-6A	Antimony	< 2.1	NJ-
PPG174-CCC02	JC19782-7A	Antimony	< 2.2	NJ-

Key:

mg/Kg – milligrams per kilogram

< –The analyte was analyzed for but was not detected above the stated reporting limit.

NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC19782

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the seven soil samples and one QC batch for the field blank.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | √ Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was detected in each of the 7 soil samples analyzed in SDG JC19782, with all sample Cr+6 results less than 12 mg/kg, all values below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time, method blanks, as well as matrix spike recoveries. No QC requirements were exceeded.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibrations demonstrated acceptable correlation coefficients (“r”) with values of 0.99932 for the soil samples analysis, as well as 0.99989 for the aqueous fraction, values greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 100.6% to 100.8% for the QC batch associated with the analysis of 7 soil samples, and 99.0 and 101.7% for the aqueous fraction, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg), the continuing calibration blanks, or the field blank (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The matrix spike and matrix spike duplicate (MS/MSD) recoveries for hexavalent chromium were all within QC limits of 75 - 125% for PPG sample JC19782-7 associated with the soil samples, such that no soil sample results were qualified for matrix spike recoveries, thereby indicating acceptable analytical accuracy in the ability to recover Cr+6 in the associated sample matrices, as demonstrated in Table 4.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC19782

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97470 ¥	JC19782-7	Cr ⁺⁶ , soluble	75.1 %	----	----
GP97470 ¥	JC19782-7	Cr ⁺⁶ , insoluble	96.8 %	----	----
GP97470 ¥	JC19782-7	Cr ⁺⁶ , post-digestion spike	96.3 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium ¥ – The samples associated with QC Batch GP97470 consist of JC19782-1 through -7 (inclusive).					

Because of the acceptable MS recoveries, no Cr+6 results required qualification in the soil sample analysis.

Duplicate Analysis (QC Limit: aqueous ≤ 20 %RPD; ≤ 35 %RPD soils)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from sample JC19782-7 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-CCC02) was 2.6%RPD, a value below the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (9.2%RPD) and pH (3.1%RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 were below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 90.3% and 93.5% associated with the soil samples and 100% for the aqueous matrix, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)
 C = Wet weight of sample (kg)
 D = % Solids/100
 E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-CCC02 (JC19782-7) was listed as 11.6 mg/kg on the reporting form and 0.2716 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.2716 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00253 \text{ Kg} \times 92.3/100} = \frac{0.02716}{0.0023352} = 11.6307 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 11.6 \text{ mg/kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 11.6 mg/kg for Sample PPG174-CCC02 was correctly reported. This was the highest detected Cr+6 concentration of the seven detected results for the 7 soil samples of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

All seven soil samples were observed to fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentrations are also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total chromium

concentrations are all less than 600 mg/kg, with most below 65 mg/kg, thereby making it less likely that Cr+6 concentrations would increase to any significant degree as observed in many other PPG data packages with total chromium concentrations below 600 mg/kg.

Summary for Hexavalent Chromium Analysis – SDG JC19782

Since the QC requirements were met in the soil samples and field blank analyses, no Cr+6 results were subject to qualification.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

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US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

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DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC19782/JC19782A

- 1. Were the appropriate sample preservation requirements met?..... Yes No
- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.
- 3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.
- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.
- 5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The nickel and vanadium results in Samples JC19782-1A and JC19782-3A exceeded the respective IGWSSL of 48 mg/kg and SRS of 78 mg/kg.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.
- 7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No

9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**

If "yes", please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds
of concern at the site met? Yes **No**

12. Were the QC Summary Forms reviewed?..... **Yes** No

13. Internal Standards acceptable..... **Yes** No

14. MS/MSD acceptable..... Yes **No**

15. Calibration summaries acceptable..... **Yes** No

16. Serial dilutions acceptable..... **Yes** No

17. Inorganic duplicates acceptable..... **Yes** No

18. LCS recovery acceptable..... **Yes** No

19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2 and 4 for QC details. Qualified sample results are presented in Table 3 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC19883/JC19883A
Sample Dates: May 9, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: June 15, 2016

This data validation (DV) report presents the data review and result qualifications for seven (7) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on May 9, 2016, for sample delivery group (SDG) JC19883, as well as JC19883A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC19883 and JC19883A were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 7 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC19883-1A through JC19883-7A (inclusive)
- Hexavalent chromium ("NJ-") in Samples JC19883-1 through JC19883-7 (inclusive)
- Hexavalent chromium ("*NJ-") in reanalysis samples JC19883-1R through JC19883-7R (inclusive)

No other sample results in SDG JC19883A and JC19883 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except the nickel result in Sample JC19883-6A, which exceeded the IGWSSL of 48 milligrams per kilogram (mg/kg), while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Tables 3 and 7 of this DV report.

Sample Receipt

The seven (7) post-excavation soil samples collected May 9, 2016, were received intact and appropriately preserved the same day, May 9, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 3.8 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC19883A and JC19883

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-RR-SW03	JC19883-1A	5/9/2016	Soil	Metals
PPG174-RR-B02	JC19883-2A	5/9/2016	Soil	Metals
PPG174-RR-SW04	JC19883-3A	5/9/2016	Soil	Metals
PPG174-MAIN-B09	JC19883-4A	5/9/2016	Soil	Metals
PPG174-MAIN-B010	JC19883-5A	5/9/2016	Soil	Metals
PPG174-MAIN-SW06	JC19883-6A	5/9/2016	Soil	Metals
PPG174-MAIN-B06R	JC19883-7A	5/9/2016	Soil	Metals
PPG174-RR-SW03	JC19883-1	5/9/2016	Soil	Cr+6
PPG174-RR-B02	JC19883-2	5/9/2016	Soil	Cr+6
PPG174-RR-SW04	JC19883-3	5/9/2016	Soil	Cr+6
PPG174-RR-SW04	JC19883-3RT	5/9/2016	Soil	TOC, SS, Fe2+
PPG174-MAIN-B09	JC19883-4	5/9/2016	Soil	Cr+6
PPG174-MAIN-B010	JC19883-5	5/9/2016	Soil	Cr+6
PPG174-MAIN-SW06	JC19883-6	5/9/2016	Soil	Cr+6
PPG174-MAIN-B06R	JC19883-7	5/9/2016	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting the metals data is numbered JC19883A, while the data package for the hexavalent chromium analyses is numbered JC19883. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC19883 together with the supplemental total organic carbon (TOC), sulfide screen and ferrous iron. The data for the five target metals were validated in this DV report, as were the hexavalent chromium, TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC19883A and JC19883 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC19883A

The data validation of the metals analytical data in SDG JC19883A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 7 post-excavation soil samples were analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 7 samples of SDG JC19883A, the nickel result in Sample JC19883-6A exhibited a concentration above the IGWSSL of 48 mg/kg for nickel. All other results were less than the respective IGWSSL and SRS limits.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP93619 indicating possible matrix interference and/or sample non-homogeneity. All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

The exceptions consisted of the 0% recovery of antimony and thallium, in CRID1 at 9:11 and CRID2 at 15:19 in analytical sequence MA39374 associated with the seven soil samples, while the vanadium recovery in CRID1 was 135%. However, the soil sample results were not affected because the reporting limits for each of these three analytes are above the respective affected range where results may be subject to qualification. The affected ranges are approximately 0 – 0.75 mg/kg for antimony and 0 – 0.5 mg/kg for thallium and vanadium where the corresponding reporting limits are approximately 2, 1 and 5 mg/kg, respectively. Vanadium was detected in each of the 7 samples at concentrations considerably above the affected range. Thus, no sample results required qualifications for calibration issues.

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC19883A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%Relative Percent Difference [RPD])

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for PPG QC batch sample JC19883-3A, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N”

to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits, including those of QC Batch GP93619.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93619 Ω	JC19883-3A	Antimony	49.1 %	49.5 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ω – The samples associated with QC Batch MP93619 consist of JC19883-1A through -7A (inclusive).						

The antimony results in the seven affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 0.9 – 4.4%RPD for soil samples for the batch QC sample associated with the soil sample analysis with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 93.5% - 98.5% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The serial dilution results associated with the soil samples ranged 0 – 0.7 percent difference (%D), values considerably below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The post-excavation soil sample analytical results for the samples of SDG JC19883A were found to be compliant with the analytical methods for the analysis of metals in the 7 post-excavation soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP93619 associated with the seven soil samples: JC19883-1A through JC19883-7A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JC19883A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-RR-SW03	JC19883-1A	Antimony	< 2.2	NJ-
PPG174-RR-B02	JC19883-2A	Antimony	< 2.4	NJ-
PPG174-RR-SW04	JC19883-3A	Antimony	< 2.3	NJ-
PPG174-MAIN-B09	JC19883-4A	Antimony	< 2.3	NJ-
PPG174-MAIN-B010	JC19883-5A	Antimony	< 2.4	NJ-
PPG174-MAIN-SW06	JC19883-6A	Antimony	< 2.1	NJ-
PPG174-MAIN-B06R	JC19883-7A	Antimony	< 2.2	NJ-

Key:
mg/Kg - milligrams per kilogram
< –The analyte was analyzed for but was not detected above the stated reporting limit.
NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC19883

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the seven post-excavation soil samples. The soil samples were re-analyzed in a second QC batch in SDG JC19883.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Data qualifiers

Hexavalent chromium was detected in six of the seven post-excavation soil samples analyzed in SDG JC19883, with all sample Cr+6 results less than 10 mg/kg, all values below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike, post spike and pH adjusted post spike recoveries in QC Batch GP97515 were outside control limits, as were the soluble and post spike recoveries in reanalysis QC Batch GP97706. The RPD value for the duplicate analysis in the re-analysis QC Batch GP97706 was above control limits due to possible sample non-homogeneity. There was not a good agreement between the sample and 1:5 dilution in both analyses. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99994 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 103.1% to 103.3% for the QC batch associated with the analysis of 7 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP97515 associated with the 7 soil samples of this SDG, while the post-digestion spike and pH-adjusted post spike recoveries were below the QC limits of 85-115%, as presented below in Table 4. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP97515 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. The insoluble MS recovery was within QC limits.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC19883

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97515 ¥	JC19883-3	Cr ⁺⁶ , soluble	58.5 %	NJ-	Low
GP97515 ¥	JC19883-3	Cr ⁺⁶ , insoluble	84.5 %	----	----
GP97515 ¥	JC19883-3	Cr ⁺⁶ , post-digestion spike	73 %	NJ-	Low
GP97515 ¥	JC19883-3	Cr ⁺⁶ , pH-adjusted post spike	73 %	NJ-	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. ¥ – The samples associated with QC Batch GP97515 consist of JC19883-1 through -7 (inclusive).					

Because the soluble MS recovery was between 50% and 75%, a QC range where DV guidelines recommend qualifying the associated sample results as estimated values flagged with “J” due to a potential low bias (NJDEP, 2009), the Cr+6 results for the affected samples were qualified for low spike recoveries and flagged with “NJ-”, as tabulated below in Table 7, together with the qualified results from the re-analysis of this QC batch.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JC19883-3 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-RR-SW04) was 0.0%RPD, a value below the 20%RPD laboratory QC limit, as well as below the 35%RPD DV advisory QC limit for technical review of soil sample data (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (8.6%RPD) and pH (1.8%RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 were below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 84.8% and 93.2% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as serial dilution is not a requirement of the analytical method and serial dilution is not addressed in DV guidelines (NJDEP, 2009). A 1:5 dilution was actually performed and there was poor agreement between the sample and the diluted aliquot. However, even though the results may be considered for qualification, the Cr+6 results are already qualified as estimated values and are flagged with “NJ-” for the low spike recoveries.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-RR-B02 (JC19883-2) was listed as 9.1 mg/kg on the reporting form and 0.1866 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.1866 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00248 \text{ Kg} \times 82.4/100} = \frac{0.01866}{0.0020435} = 9.1313 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 9.1 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 9.1 mg/kg for Sample PPG174-RR-B02 was correctly reported. This was the highest detected Cr+6 concentration of the six detected results for the 7 soil samples of the initial analysis in this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

All seven soil samples were observed to clearly fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentrations are also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total chromium concentrations are all less than 320 mg/kg, thereby making it highly unlikely that Cr+6 concentrations would increase to any significant degree, as observed in many other PPG data packages with total chromium concentrations below 600 mg/kg.

A review of the chromium to Cr+6 ratios for the samples of SDG JC19883 revealed that the Cr:Cr+6 ratios of samples falling within the “reducing” zone exhibited Cr:Cr+6 ratios ranging 10 to 80, similar to those ratios observed in PPG SDG JC21931. Review of the chromium and Cr+6 results of the many analyzed soil samples for the PPG project suggests that generally the Cr:Cr+6 ratios tend to generally fall above a ratio of 20 to 1, which is not inconsistent with other studies in sites within New Jersey (Paustenbach, et al., 1991).

Hence, based on the sample total chromium and Cr+6 concentrations, it is highly unlikely that any of the affected samples including those in the “reducing” zone would approach the SCC for Cr+6 of 20 mg/kg due to limitation created by the relatively low total chromium concentrations available for potential oxidation.

Cr+6 Re-analyses in SDG JC19883

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the re-analysis batch consisting of 7 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC19883-1R through -7R in QC Batch GP97706, including the calibrations ($r = 0.99996$, 93.7 – 94.2% CCV Recoveries), QC blanks, and blank spike analysis (91.7% – 92.3%). The soluble MS recovery was lower in the reanalysis, falling below 50% and still below QC limits, while the post-digestion spike and pH-adjusted post spikes were slightly higher, but the post spike was still below QC limits, as detailed below. There was not a good agreement between the samples and the 1:5 dilution.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the soluble and insoluble MS recoveries in QC Sample JC19883-3R were lower than the initial analysis, while the post-digestion spike and pH-adjusted post spike recoveries were higher in the reanalysis, as observed below in Table 5. The insoluble MS and pH-adjusted post spike recoveries in JC19883-3R were within the respective QC limits.

Table 5. Hexavalent Chromium Re-analysis MS Recovery Results – JC19883

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97706 Ж	JC19883-3R	Cr ⁺⁶ , soluble	42.9 %	NJ-	Low
GP97706 Ж	JC19883-3R	Cr ⁺⁶ , insoluble	77.8 %	----	----
GP97706 Ж	JC19883-3R	Cr ⁺⁶ , post-digestion spike	81 %	NJ-	Low
GP97706 Ж	JC19883-3R	Cr ⁺⁶ , pH-adjusted post spike	85 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
 MS – Matrix spike
 Cr⁺⁶ – Hexavalent chromium
 NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.
 Ж – The samples associated with QC Batch GP97706 consist of JC19883-1R through -7R (inclusive).

Since the soluble MS recovery in QC Batch GP97706 was still below the QC limits (75-125%), the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” for a potential low bias in the ability to recover Cr+6 in this QC batch.

The Cr+6 results qualified for low spike recoveries are tabulated below in Table 7, together with the qualified results from the initial analysis of this set of samples. Since the MS recovery was < 50% in the re-analysis, the results were subject to rejection (NJDEP, 2009), however, based on the review of the Eh/pH data and other factors, as explained below, the five detected and two non-detected Cr+6 results in the re-analysis of the soil samples of SDG JB19883 were qualified as estimated results, and flagged with “*NJ-”. The post-digestion spike recovery value in the re-analysis is 85%, just meeting the QC limit. The qualified Cr+6 results of the reanalysis are presented below in Table 7 together with the results of the initial Cr+6 results.

Duplicate Sample Analysis (≤ 35%RPD soils)

The duplicate analysis was performed on one set of duplicate soil sample aliquots. The %RPD value (89.7%RPD) for duplicate samples in QC Batch GP97706 was well above the QC limit of 35%RPD for soil samples (US EPA, 2010; AECOM, 2010), as listed in Table 6. A possible cause of the observed differences between the duplicate results may be attributable to sample non-homogeneity. Consequently, since the duplicate Cr+6 analysis exceeded the QC limit, the Cr+6 results for the re-analysis of the seven soil samples results are qualified as estimated values with an indeterminate bias and are to be flagged with the qualifier “*J” (NJDEP, 2009), as presented below in Table 6.

Table 6. Duplicate Analysis Results Outside QC Limits

QC Batch	QC Sample	Analyte	Original Result (mg/kg)	Duplicate (mg/kg)	Difference	DV Qualifier
GP97706 Ж	JB19883-3R	Cr+6	4.2	1.6	89.7 %RPD	*J

QC Limit is 35%RPD
 mg/kg = milligrams per kilogram
 * – Duplicate analysis not within control limits; indeterminate bias direction.
 J – The reported result is an estimated value.
 Ж – The samples associated with QC Batch GP97706 consist of JC19883-1R through -7R (inclusive)

Since the Cr+6 duplicate analysis exceeded the QC limit, the associated samples are subject to qualification as estimated values and are to be flagged with ‘*J’ for to the possible variability in the analytical precision. These Cr+6 results qualified for both low MS recoveries and the variability in the duplicate analysis are flagged with “*NJ-” in Table 7, below, for samples with laboratory ID numbers ranging JC19883-1R through -7R.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC19883-3RT (PPG174-RR-SW04), a QC samples which was analyzed twice with detected Cr6+ concentrations of 1.7 and 4.2 mg/kg for these analyses, values well below the SCC of 20 mg/kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. The total organic carbon (TOC) analysis was performed within the 14-day analytical holding time. In accordance with the method, these analyses were performed on the sample experiencing the low spike recoveries. A concentration of total organic carbon (44,800 mg/kg) and the ferrous iron (Fe+2) with a result of 0.22 % were detected in the QC sample in JC19883-3RT, thereby indicating the likely presence of a “reducing” soil matrix in the soil sample, as suggested by the presence of this soil sample below the Eh-pH phase line, as are the other six soil samples of this SDG.

The “reducing” conditions in the soil matrix appear supported by the detected TOC concentration and the detected Fe⁺² data in support of the results of the Eh-pH analyses.

Summary for Hexavalent Chromium Analysis – SDGs JC19883

The qualified soil sample results from the initial Cr+6 analysis in SDG JC19883 are presented below in Table 7 alongside those qualified results obtained from the reanalysis of the samples. Both sets of analytical Cr+6 results for samples JC19883-1 through -7 and their reanalysis are still both qualified as estimated values (NJ-) due to a potential low bias, although the soluble MS recoveries of the second analysis exhibited lower recoveries in the re-analyses that were performed within the 30-day holding time. The Cr+6 results of the re-analysis are also flagged with “*” because of the potential variability in the analytical precision as suggested by the results in the duplicate analysis. The Cr+6 concentrations determined during the re-analysis of samples in SDG JC19883 differ slightly from those of the initial analysis, but with the exception of the 20.0 mg/kg result for JC19883-7R and 18.9 mg/kg in JC19883-2R, all are still well below the SCC of 20 mg/kg.

Table 7. Comparison of Qualified Cr+6 Results in JC19883 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC19883 Result (mg/kg)	DV Qualifier	JC19883-R Results (mg/kg)	DV Qualifier
PPG174-RR-SW03	JC19883-1	Cr+6	7.9	NJ-	4.9	*NJ-
PPG174-RR-B02	JC19883-2	Cr+6	9.1	NJ-	18.9	*NJ-
PPG174-RR-SW04	JC19883-3	Cr+6	1.7	NJ-	4.2	*NJ-
PPG174-MAIN-B09	JC19883-4	Cr+6	1.3	NJ-	< 0.47	*NJ-
PPG174-MAIN-B010	JC19883-5	Cr+6	< 0.49	NJ-	< 0.49	*NJ-
PPG174-MAIN-SW06	JC19883-6	Cr+6	8.2	NJ-	8.1	*NJ-
PPG174-MAIN-B06R	JC19883-7	Cr+6	6.3	NJ-	20.0	*NJ-

mg/kg - milligrams per kilogram
 < –The analyte was analyzed for but was not detected above the stated reporting limit.
 * – Duplicate analysis not within control limits; indeterminate bias direction.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is

Client ID	Laboratory Sample ID	Analyte	JC19883 Result (mg/kg)	DV Qualifier	JC19883-R Results (mg/kg)	DV Qualifier
estimated and may be biased low.						

Professional judgement was applied in qualifying the Cr+6 results in both analyses as estimated values (NJ-) due to a potential low bias, as suggested by the MS results tabulated above in Table 4 and Table 5. Because the soluble MS recovery in the initial analysis was above 50%, the results are clearly subject to qualification as estimated values (NJDEP, 2009).

Although the soluble MS recovery in the re-analysis QC Batch GP97706 was less than 50% (42.9%) and the results subject to rejection (NJDEP, 2009), the associated detected Cr+6 sample concentrations in the re-analysis were only qualified as estimated values and flagged with “*NJ-“, rather than be rejected, because the insoluble recovery (77.8%) was within QC limits, a **data usability** approach previously discussed with Mr. Joseph Sanguiliano of the NJDEP. Inorganic data validation guidelines do not recommend rejection of detected sample results (US EPA, 2014) and Cr+6 was detected in six of the seven soil samples of the initial analysis and five in the re-analysis. The non-detect Cr+6 results for JB19883-4R and -5R were also not rejected and only qualified as estimated reporting limits (< 0.47 *NJ- and < 0.49 *NJ-) because the total chromium concentrations were both less than 14 mg/kg and are highly unlikely to exceed the SCC of 20 mg/kg because the samples were collected from a “reducing” soil environment, which does not favor oxidation of chromium to Cr+6, such that conversion to Cr+6 is not anticipated. Additionally, the insoluble MS recovery was within QC limits for both analyses and may be a better representation of the ability of the analysis to recover Cr+6 from the soil matrix than the soluble MS recovery result, as suggested by the results of the many PPG soil sample analyses. The low MS recovery in the soluble matrix spike analysis suggests a potential low bias in the ability to recover Cr+6 in this QC batch. Consequently, the soil samples of this QC batch are qualified as estimated values and flagged with “*NJ-” in the reanalysis data set, as represented in Table 7.

Professional judgement was applied in qualifying, not rejecting, the Cr+6 results in the reanalysis of the soil samples of SDG JC19883. Because inorganic data validation guidelines do not recommend rejection of detected sample results (USEPA, 2014), the detected Cr+6 concentrations in five samples were qualified (“*NJ-”). The non-detect Cr+6 result in the two samples exhibiting non-detect results for Cr+6 in the reanalysis associated with the 42.9% soluble MS recovery had corresponding total chromium results less than 14 mg/kg, thereby making it highly improbable that sample Cr+6 would approach the SCC of 20 mg/kg. Due to the low chromium concentrations in the two samples exhibiting non-detect Cr+6 results, the acceptable insoluble MS recovery in both the initial analysis and the re-analysis, and the “reducing” soil conditions, it was judged appropriate to not reject the Cr+6 results of the re-analysis primarily because of the low chromium content of the samples in a “reducing” soil matrix. Hence, due to the absence of sufficient chromium that could be oxidized to Cr+6 it is unlikely that Cr+6 concentrations could approach the SCC of 20 mg/kg, the non-detect results in JC19883-4R and -5R are considered usable and were not rejected by considering data usability concepts.

Professional judgement was applied in not rejecting the Cr+6 result in the re-analysis sample JC19883-5R, because it was identical to the non-detect result in the initial analysis where DV guidelines recommend qualification of the associated results when MS recoveries fall between 50-75% (NJDEP, 2009). Because the results in the analyses of JC19883-5 are identical and contain only a very low corresponding total chromium concentration of 12.9 mg/kg, it was judged unwarranted to reject the Cr+6 result of the re-analysis when the initial result is evidently subject to qualification.

Although the samples were re-analyzed within the 30-day holding time, the Cr+6 concentrations differed slightly upon reanalysis. However, except for the increased Cr+6 results for samples JC19883-2R and -7R in the reanalysis, despite the lower spike recoveries, the Cr+6 sample results exhibited Cr+6 values considerably below the SCC of 20 mg/kg, consistent with the redox state of the sample's soil environment.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
*	Duplicate analysis not within control limits; indeterminate bias direction.
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

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US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC19883/JC19883A

- 1. Were the appropriate sample preservation requirements met?..... Yes No

- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

- 3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

- 5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The nickel result in Sample JC19883-6A exceeded the IGWSSL of 48 mg/kg. The 20 mg/kg Cr+6 result in re-analysis Sample JC19883-7R was detected at the SCC of 20 mg/kg.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

- 7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.
Not applicable
10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... Yes **No**
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2, 4, 5, and 6 for QC details. Qualified sample results are presented in Tables 3 and 7 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC19973/JC19973A
Sample Dates: May 10, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: May 31, 2016

This data validation (DV) report presents the data review and result qualifications for five (5) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on May 10, 2016, for sample delivery group (SDG) JC19973, as well as JC19973A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC19973 and JC19973A were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 5 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Hexavalent chromium (“*NJ-”) in Samples JC19973-2 through JC19973-6 (inclusive)
- Hexavalent chromium (“*NJ-”) in re-analysis samples JC19973-2R through JC19973-6R (inclusive)

No other sample results in SDG JC19973A and JC19973 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except the nickel result in Sample JC19973-6A, which exceeded the IGWSSL of 48 milligrams per kilogram (mg/kg), while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) in the respective SDGs, except for the re-analysis sample JC19973-3R. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Table 6 of this DV report.

Sample Receipt

The five (5) post-excavation soil samples collected May 10, 2016, were received intact and appropriately preserved the same day, May 10, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 3.7 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC19973A and JC19973

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-RR-B03	JC19973-2A	5/10/2016	Soil	Metals
PPG174-MAIN-CC03	JC19973-3A	5/10/2016	Soil	Metals
PPG174-MAIN-CC04	JC19973-4A	5/10/2016	Soil	Metals
PPG174-MAIN-SW07	JC19973-5A	5/10/2016	Soil	Metals
PPG174-MAIN-SW08	JC19973-6A	5/10/2016	Soil	Metals
PPG174-RR-B03	JC19973-2	5/10/2016	Soil	Cr+6
PPG174-MAIN-CC03	JC19973-3	5/10/2016	Soil	Cr+6
PPG174-MAIN-CC04	JC19973-4	5/10/2016	Soil	Cr+6
PPG174-MAIN-SW07	JC19973-5	5/10/2016	Soil	Cr+6
PPG174-MAIN-SW08	JC19973-6	5/10/2016	Soil	Cr+6
PPG174-MAIN-SW08	JC19973-6RT	5/10/2016	Soil	TOC, SS, Fe2+
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting the metals data is numbered JC19973A, while the data package for the hexavalent chromium analyses is numbered JC19973. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC19973 together with the supplemental total organic carbon (TOC), sulfide screen and ferrous iron. The samples data were validated for the five target metals (antimony, chromium, nickel, thallium, and vanadium), as were the hexavalent chromium data, and supplemental TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC19973A and JC19973 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).

- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC19973A

The data validation of the metals analytical data in SDG JC19973A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | √ Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 5 post-excavation soil samples were analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 5 samples of SDG JC19973A, the

nickel result in Sample JC19973-6A exhibited a concentration above the IGWSSL of 48 mg/kg for nickel. All other results were less than the respective IGWSSL and SRS limits.

Laboratory Case Narrative

The case narrative stated that the relative percent difference (RPD) for the serial dilution analysis for thallium was outside control limits in QC Batch MP93631, however, the percent difference (%D) result was acceptable due to the low initial sample concentration (< 50 times instrument detection limit [IDL]). All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and "low calibration check standard" ("CRI" standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exception.

The exception consisted of the 163.3% recovery of antimony in CRID2 at 11:06 in analytical sequence MA39382 preceding the five soil samples. However, the soil sample results for antimony were not affected because there is no positive bias in a non-detect result. Antimony was not detected in any of the five associated soil samples. Thus, no sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC19973A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis (QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for the five target metals were within the QC limits of 75 - 125% for non-client QC batch sample JC19810-2, such that no sample metals results required qualification for matrix spike recovery results.

The metals results in the five associated soil samples are not subject to qualification and the MS recovery results demonstrate acceptable accuracy.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples (US EPA, 2010), with values ranging from 0.5 – 2.5%RPD for soil samples for the

batch QC sample associated with the soil sample analysis with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 98.0% - 101.5% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative stated that the RPD serial dilution result for thallium was outside control limits in QC Batch MP93631; however, the percent difference (%D) result was acceptable due to a low initial sample concentration (< 50 times IDL). The remaining three serial dilution results associated with the soil samples ranged from 0.0 – 3.9%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC19973A were found to be compliant with the analytical methods for the analysis of metals in the 5 soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, such that no soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC19973

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the five post-excavation soil samples.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was detected in each of the five post-excavation soil samples analyzed in SDG JC19973 and the re-analysis, with all sample Cr+6 results below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg, except re-analysis sample JC19973-3R (20.3 mg/kg).

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike and post spike recoveries in QC Batch GP97542 were outside control limits, while the soluble MS recovery was outside control limits as well as in reanalysis QC Batch GP97577. The case narrative also stated that the pH adjusted post spike was low in the initial Cr+6 analysis, however, the 104.63% recovery is well within the 85-115% QC limits. The RPD value for the duplicate analysis in the analysis QC Batch GP97542 and the reanalysis in GP97577 were above control limits due to possible sample non-homogeneity. There was not a good agreement between the sample and 1:5 dilution in the initial analysis. The ferrous iron and sulfide screen test were analyzed after completion of Cr+6 testing (outside of normal hold time) in order to provide more information about the possible impact of the sample matrix on Cr+6 recoveries. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99990 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 95.0% to 95.3% for the QC batch associated with the analysis of 5 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP97542 associated with the 5 soil samples of this SDG, and the post-digestion spike recovery was below the corresponding QC limits of 85-115%, as presented below in Table 2. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP97542 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. The insoluble MS and pH-adjusted spike recoveries were within the respective QC limits.

Table 2. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC19973

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97542 ¥	JC19973-6	Cr ⁺⁶ , soluble	41.3 %	NJ-	Low
GP97542 ¥	JC19973-6	Cr ⁺⁶ , insoluble	94.2 %	----	----
GP97542 ¥	JC19973-6	Cr ⁺⁶ , post-digestion spike	78.7 %	NJ-	Low
GP97542 ¥	JC19973-6	Cr ⁺⁶ , pH-adjusted post spike	104.63 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium					

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. ¥ – The samples associated with QC Batch GP97542 consist of JC19973-2 through -6 (inclusive).					

The detected Cr+6 results in the 5 affected soil samples were qualified for low spike recoveries to be flagged with “NJ-” (US EPA, 2014), as tabulated below in Table 6, together with the qualified results from the re-analysis of this QC batch. Although the soluble MS recovery was below the 50% criterion, below which DV guidelines for Cr+6 analysis recommend rejection of associated results (NJDEP, 2009), the results were qualified as estimated values because inorganic DV guidelines do not recommend rejection of detected concentrations (US EPA, 2014) and the insoluble MS recovery was well within QC limits.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from sample JC19973-6 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-MAIN-SW08) was 102.2%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD DV advisory QC limit for technical review of soil sample data (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (0.9%RPD) and pH (0.3 %RPD) displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was above the QC limit for soil samples, the associated sample results are subject to qualification.

Table 3. Duplicate Analysis Results Outside QC Limits

QC Batch	QC Sample	Analyte	Original Result (mg/kg)	Duplicate (mg/kg)	Difference	DV Qualifier
GP97542 ¥	JB19973-6	Cr+6	6.8	21.0	102.2 %RPD	*J
mg/kg - milligrams per kilogram QC Limit is 35%RPD. * – Duplicate analysis not within control limits; indeterminate bias direction. J – The reported result is an estimated value. ¥ – The samples associated with QC Batch GP97542 consist of JC19973-2 through -6 (inclusive)						

Since the duplicate analysis for Cr+6 had exceeded the QC limit for duplicate soil sample analysis, the associated PPG samples with laboratory ID numbers JC19973-2 through -6 (inclusive) were qualified as estimated values due to the potential variability in the analytical precision. Because the soluble MS recovery was also below QC limits, the Cr+6 results for these five soil samples are flagged with “*NJ-” as presented below in the summary table, Table 6.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 84.8% and 87.8% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as serial dilution is not a requirement of the analytical method and serial dilution is not addressed in DV guidelines (NJDEP,

2009). A 1:5 dilution was actually performed and there was poor agreement between the sample and the diluted aliquot. However, even though the results may be considered for qualification, the Cr+6 results are already qualified as estimated values and are flagged with “*NJ-” for the low spike recoveries and disparity in the duplicate analysis. No additional qualification is warranted.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-MAIN-CC03 (JC19973-3) was listed as 11.5 mg/kg on the reporting form and 0.2736 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.2736 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00260 \text{ Kg} \times 91.5/100} = \frac{0.02736}{0.0023790} = 11.5006 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 11.5 \text{ mg/kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 11.5 mg/kg for Sample PPG174-MAIN-CC03 was correctly reported. This was the highest detected Cr+6 concentration of the five detected results for the soil samples in the initial analysis of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

All five soil samples were observed to clearly fall above the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of an “oxidizing” soil environment. While Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions, the oxidizing soil conditions may contribute to oxidation of total chromium to Cr+6, provided there is sufficient chromium present in the soil.

Cr+6 Re-analysis in SDG JC19973

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the re-analysis batch consisting of 5 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC19973-2R through -6R in QC Batch GP97557, including the calibrations ($r = 0.99969$, 102.9 – 103.1% CCV Recoveries), QC blanks, and blank spike analysis (93.5% – 96.8%). The soluble MS recovery was higher in the reanalysis, increasing to a recovery above 50%, but still below QC limits, while the post-digestion spike improved to a value within QC limits, as detailed below.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the soluble, insoluble MS, and post-digestion spike recoveries improved to the degree that the soluble MS recovery was above 50%, and the insoluble MS and post spike improved to fall within the respective QC limits in QC Sample JC19973-6R, as observed below in Table 4. The insoluble MS and pH-adjusted post spike recoveries in JC19973-6R were within the respective QC limits.

Table 4. Hexavalent Chromium Re-analysis MS Recovery Results – JC19973

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97577 Ж	JC19973-6R	Cr ⁺⁶ , soluble	59.4 %	NJ-	Low
GP97577 Ж	JC19973-6R	Cr ⁺⁶ , insoluble	98.3 %	----	----
GP97577 Ж	JC19973-6R	Cr ⁺⁶ , post-digestion spike	92 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ж – The samples associated with QC Batch GP97577 consist of JC19973-2R through -6R (inclusive).					

Since the soluble MS recovery in QC Batch GP97577 improved to 59.4% but was still below the QC limits (75-125%), the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” for a potential low bias in the ability to recover Cr+6 in this QC batch, because the soluble MS recovery was between 50-75% (NJDEP, 2009). The qualified Cr+6 results of the reanalysis are presented below in Table 6 together with the results of the initial Cr+6 results.

Duplicate Sample Analysis ($\leq 35\%$ RPD soils)

The duplicate analysis was performed on one set of duplicate soil sample aliquots. The %RPD value (64.1%RPD) for duplicate samples in QC Batch GP97577 was well above the QC limit of 35%RPD for soil samples (US EPA, 2010; AECOM, 2010), as listed in Table 5. A possible cause of the observed differences between the duplicate results may be attributable to sample non-homogeneity. Consequently, since the duplicate Cr+6 analysis exceeded the QC limit, the Cr+6 results for the re-analysis of the five soil samples results are qualified as estimated values with an indeterminate bias and are to be flagged with the qualifier “*J” (NJDEP, 2009), as presented below in Table 5.

Table 5. Duplicate Analysis Results Outside QC Limits

QC Batch	QC Sample	Analyte	Original	Duplicate	Difference	DV
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			Result (mg/kg)	(mg/kg)		Qualifier
GP97577 Ж	JB19973-6R	Cr+6	15.7	30.5	64.1 %RPD	*J
QC Limit is 35%RPD; * – Duplicate analysis not within control limits; indeterminate bias direction. J – The reported result is an estimated value. Ж – The samples associated with QC Batch GP97577 consist of JC19973-2R through -6R (inclusive).						

Since the Cr+6 duplicate analysis exceeded the QC limit, the associated samples are subject to qualification as estimated values and are to be flagged with ‘*J’ for to the possible variability in the analytical precision. These Cr+6 results qualified for both low MS recoveries and the variability in the duplicate analysis are flagged with “*NJ-” in Table 6, below, for samples with laboratory ID numbers ranging JC19973-2R through -6R.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC19973-6RT (PPG174-MAIN-SW08), a QC sample which was analyzed twice with detected Cr+6 concentrations of 6.8 and 15.7 mg/kg, values below the SCC of 20 mg/kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. The total organic carbon (TOC) analysis was performed within the 14-day analytical holding time. The MS recovery in the QC sample associated with JC19973-6RT was above QC limits, however, the TOC result in JC19973-6RT was not subject to qualification because the initial TOC concentration in the batch QC sample was more than 4 times the spike value, potentially causing masking of the spike. Hence, the TOC result was not qualified as an estimated value.

In accordance with the method, these supporting analyses were performed on the sample experiencing the low spike recoveries. A concentration of total organic carbon (28,400 mg/kg) and the ferrous iron (Fe+2) with a result of 0.27 % were detected in the QC sample in JC19973-6RT, thereby indicating the likely presence of a “reducing” soil matrix in the soil sample. However, this observation is in contrast to the results of the pH and redox analyses where Sample JC19973-6 was observed to fall above the Eh-pH phase line, thereby representing an “oxidizing” soil matrix.

The “reducing” conditions in the soil matrix suggested by the detected 2.8% TOC concentration and the detected Fe⁺² data, although in contrast to the results of the Eh-pH analyses, may be the reason the total chromium concentration may not have contributed to a greater Cr+6 concentration in Sample JC19973-6.

Summary for Hexavalent Chromium Analysis – SDG JC19973

The qualified soil sample results from the initial Cr+6 analysis in SDG JC19973 are presented below in Table 6 alongside those qualified results obtained from the reanalysis of the samples. Both sets of analytical Cr+6 results for samples JC19973-2 through -6 and their reanalysis are still both qualified as estimated values (*NJ-) due to a potential low bias in the ability to recover Cr+6 from the soil matrix and potential variability in the analytical precision, although the soluble MS recoveries of the second analysis exhibited a higher recovery in the re-analysis that was performed within the 30-day holding time. The Cr+6 results of the initial and re-analysis are flagged with “*” because of the potential variability in the analytical precision as suggested by the results in the

duplicate analyses. The Cr+6 concentrations determined during the re-analysis of samples in SDG JC19973 differ slightly from those of the initial analysis, but with the exception of the 20.3 mg/kg result for JC19973-3R, all are still below the SCC of 20 mg/kg.

Table 6. Comparison of Qualified Cr+6 Results in JC19973 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC19973 Result (mg/kg)	DV Qualifier	JC19973-R Results (mg/kg)	DV Qualifier
PPG174-RR-B03	JC19973-2	Cr+6	11.3	*NJ-	9.2	*NJ-
PPG174-MAIN-CC03	JC19973-3	Cr+6	11.5	*NJ-	20.3	*NJ-
PPG174-MAIN-CC04	JC19973-4	Cr+6	10.4	*NJ-	13.9	*NJ-
PPG174-MAIN-SW07	JC19973-5	Cr+6	6.9	*NJ-	14.7	*NJ-
PPG174-MAIN-SW08	JC19973-6	Cr+6	6.8	*NJ-	15.7	*NJ-

mg/kg - milligrams per kilogram
 * – Duplicate analysis not within control limits; indeterminate bias direction.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
 NJ+ – The matrix spike sample recovery in the associated QC sample is above QC limits; the result is estimated and may be biased high.

Although the Cr+6 results of the initial analysis may be subject to rejection because the soluble MS recovery of 41.3% was below the 50% criterion where the Cr+6 DV guidelines recommend rejection of associated sample results (NJDEP, 2009), the Cr+6 results in both the initial and re-analysis were qualified as estimated values and not rejected due to a data usability approach and professional judgement based on the following considerations.

First of all, Cr+6 concentrations were detected in each of the five post-excavation samples, both in the initial analysis and the re-analysis, and inorganic data validation guidelines do not recommend rejection of detected results (USEPA, 2014). Both analyses were performed within the 30-day analytical holding time. Because the 59.4% soluble MS recovery in the re-analysis falls between 50-75%, the associated sample Cr+6 results of the re-analysis are qualified as estimated values (“flag the data as estimated with J”) based on current data validation guidance for Cr+6 analysis (NJDEP, 2009). In consideration of the extensive amount of Cr+6 analyses performed at various PPG sites, it appears that the ability to recover Cr+6 from PPG soil samples is correlated more with the insoluble MS recoveries than results of the soluble MS recoveries. Because the insoluble MS recoveries were both well within QC limits in the initial, as well as the re-analysis, this provides additional support for qualifying, and not rejecting, the Cr+6 results of the initial analysis.

While the Cr+6 results in most of the samples increased in the re-analysis, presumably due to improved MS recoveries, including the insoluble MS, soluble MS, as well as post-digestion spike recovery, the increase in Cr+6 results may also be attributable, in part, to sample non-homogeneity such as the increase in JC19973-3R, but also the decrease in JC19973-2R.

Although the samples were re-analyzed within the 30-day holding time, the Cr+6 concentrations appear to differ slightly upon reanalysis. While the results of the re-analysis generally appear to have increased due in part to the increased MS recoveries of the reanalysis, professional judgement was applied in not rejecting the Cr+6 results of the initial analysis because Cr+6 was detected in each sample for both analyses. Because project activities tend to be based on the higher of two analytical results for a particular sample, it was judged unnecessary to reject the initial results because they are detected results, though generally less than those of the re-analysis that are rightly subject to qualification and not rejection. The results of the initial analysis do, to

some extent, support the observed results of the re-analysis and, therefore, are considered to provide support and verification of the results detected in the re-analysis.

The Cr+6 sample results, except the re-analysis result of JC19973-3R, exhibited a Cr+6 values considerably below the SCC of 20 mg/kg, despite the presumed presence of an “oxidizing” soil condition suggested by the Eh-pH phase diagram. However, the contradictory information observed for the supplemental TOC and ferrous iron results for JC19973-6RT may have contributed somewhat to limiting oxidation of chromium to Cr+6 in this sample, JC19973-6.

There were five soil samples that were observed to fall above the Eh-pH phase diagram line in JC19973. The chromium to Cr+6 ratios (Cr:Cr+6) in these five samples representing “oxidizing” conditions ranged from approximately 6 – 75, which are not unlike those ratios of 14 – 38 observed in the five “oxidizing” samples of JC18120, where the Cr+6 results were not subject to qualification because each of the MS recoveries were within QC limits, thereby demonstrating acceptable accuracy and ability to recover Cr+6 from the soil samples under oxidizing conditions. Since the Cr:Cr+6 ratios of samples experiencing low recoveries in JC19973 were not unlike those for oxidizing soils with acceptable recoveries in JC18120, this further supports the professional judgement to not reject the Cr+6 results of the initial analysis in JC19973 that are considered usable within the context of the qualifications.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
*	Duplicate analysis not within control limits; indeterminate bias direction.
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***"National Functional Guidelines for Inorganic Superfund Data Review"***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC19973/JC19973A

- 1. Were the appropriate sample preservation requirements met?..... Yes No
- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.
- 3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.
- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.
- 5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The nickel result in Sample JC19973-6A exceeded the IGWSSL of 48 mg/kg. The Cr+6 result (20.3 mg/kg) in re-analysis sample JC19973-3R marginally exceeded the SCC of 20 mg/kg.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.
- 7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... Yes **No**
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2, 3, 4, and 5 for QC details. Qualified sample results are presented in Table 6 of this DV report.



DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC20350/JC20350A
Sample Dates: May 16, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: June 23, 2016

This data validation (DV) report presents the data review and result qualifications for two (2) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, from May 16, 2016, for sample delivery group (SDG) JC20350, as well as JC20350A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC20350A and JC20350 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 2 collected post-excavation soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC20350-1A and JC20350-2A
- Hexavalent chromium ("NJ-") in Samples JC20350-1 and JC20350-2
- Hexavalent chromium ("NJ-") in reanalysis samples JC20350-1R and JC20350-2R
- Total organic carbon ("J") in Sample JC20350-2RT

No other sample results in SDG JC20350A and JC20350 required qualification, based on the acceptable remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) concentrations were below the Soil Cleanup Criterion (SCC) of 20 mg/kg in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Tables 3 and 6 of this DV report.

Sample Receipt

The two (2) soil samples collected May 16, 2016, were received intact and appropriately preserved the same day, May 16, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 2.8 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC20350A and JC20350

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-B11	JC20350-1A	5/16/2016	Soil	Metals
PPG174-MAIN-B12	JC20350-2A	5/16/2016	Soil	Metals
PPG174-MAIN-B11	JC20350-1	5/16/2016	Soil	Cr+6
PPG174-MAIN-B12	JC20350-2	5/16/2016	Soil	Cr+6
PPG174-MAIN-B12	JC20350-2RT	5/16/2016	Soil	TOC, SS, Fe2+
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting the metals data is numbered JC20350A, while the data package for the hexavalent chromium analyses is numbered JC20350. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC20350 together with the supplemental total organic carbon (TOC), sulfide screen and ferrous iron. The samples data were validated for the five target metals (antimony, chromium, nickel, thallium, and vanadium), as were the hexavalent chromium data, and supplemental TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC20350A and JC20350 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.

- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC20350A

The data validation of the metals analytical data in SDG JC20350A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 2 post-excavation soil samples were analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 2 soil samples of SDG JC20350A, all analyte results were below the respective IGWSSL and SRS limits.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP93769 indicating possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution result for antimony was outside control limits in QC Batch MP93769; however, the percent difference (%D) result was acceptable due to a low

initial sample antimony concentration (< 50 times instrument detection limit [IDL]). All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

The exception consisted of the 150% recovery of antimony in CRID2 at 12:16 in analytical sequence MA39421. However, CRID2 was not directly associated with the 2 soil samples of this SDG, since the results of CRID3 at 15:06, which preceded the soil sample analysis, exhibited acceptable recoveries. Additionally, the soil sample results were not subject to qualification because the non-detect result in JC20350-1A is not affected by a potential positive bias, while the detected concentration of 4.2 mg/kg in JC20350-2A is above the affected range of approximately 0-0.5 mg/kg extrapolated from another PPG SDG displaying a full metals data deliverable.

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC20350A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for a non-client QC batch sample JC20336-2, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93769 Ω	JC20336-2	Antimony	39.2 %	44.8 %	NJ-	Low

QC Limits are 75-125%;
MS – Matrix spike
MSD – Matrix spike duplicate.
NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias.
Ω – The samples associated with QC Batch MP93769 consist of JC20350-1 and JC20350-2.

The antimony results in these two affected soil samples are flagged with “NJ-” due to a potential low bias. The metals concentrations in the non-client QC sample appear to be similar to those typically observed in PPG samples; therefore, qualification of the associated antimony results was judged appropriate in this case. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 1.6 – 14.3%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated acceptable analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 94.2% - 98.9% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit ≤ 10 %D)

The case narrative stated that the RPD serial dilution result for antimony was outside control limits in QC Batch MP93769; however, the percent difference (%D) result was acceptable due to a low initial sample concentration (< 50 times IDL). The remaining four serial dilution results associated with the soil samples ranged from 0 – 7.4%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC20350A were found to be compliant with the analytical methods for the analysis of metals in the 2 soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP93769 associated with the 2 soil samples: JC20350-1 and JC20350-2. The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JC20350A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-B11	JC20350-1A	Antimony	< 2.5	NJ-
PPG174-MAIN-B12	JC20350-2A	Antimony	4.2	NJ-

Key:
 mg/kg - milligrams per kilogram
 < –The analyte was analyzed for but was not detected above the stated reporting limit.
 NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC20350

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the two post-excavation soil samples. The soil samples were re-analyzed in a second QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Data qualifiers

Hexavalent chromium was detected in both soil samples analyzed in SDG JC20350, with the sample Cr+6 result of 1.5 mg/kg being the highest concentration in the initial analysis, a result clearly below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble and insoluble matrix spike recoveries in QC Batch GP97681 were outside control limits, as well as the soluble MS recovery in reanalysis QC Batch GP98058. The RPD value for the duplicate analysis in the re-analysis QC Batch GP98058 was above control limits, but the RPD was acceptable due to low duplicate and sample concentrations. Sample JC20350-2RT was prepared outside the holding time for the total organic carbon (TOC) analysis. The ferrous iron and sulfide screen test were analyzed after completion of Cr+6 testing (outside of normal hold time) in order to provide more information about the possible impact of the sample matrix on Cr+6 recoveries. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% CCV Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99984 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 95.8% to 96.1% for the QC batch

associated with the analysis of 2 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery were below the QC limits of 75-125% for QC Batch GP97681 associated with the 2 soil samples of this SDG, as presented below in Table 4. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP97681 qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. All remaining MS recoveries were within QC limits.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC20350

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97681 ¥	JC20350-2	Cr ⁺⁶ , soluble	13.9 %	NJ-	Low
GP97681 ¥	JC20350-2	Cr ⁺⁶ , insoluble	72.5 %	NJ-	Low
GP97681 ¥	JC20350-2	Cr ⁺⁶ , post-digestion spike	85.59 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
 MS – Matrix spike
 Cr⁺⁶ – Hexavalent chromium
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
 ¥ – The samples associated with QC Batch GP97681 consist of JC20350-1 and JC20350-2.

The Cr+6 results qualified for low spike recoveries are flagged with “NJ-” (USEPA, 2014), as tabulated below in Table 6, together with the qualified results from the re-analysis of this QC batch. Although the soluble MS recovery was below 50%, a value below which DV guidelines recommend rejection of associated Cr+6 results (NJDEP, 2009), the results were qualified, rather than rejected, based on data usability concepts for reasons discussed below in the summary section.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from PPG sample JC20350-2 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-MAIN-B12) was 8.7%RPD, a value below the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (2.7%RPD) and pH (1.0%RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 90.5% and 89.0% associated with the soil sample analysis, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-MAIN-B11 (JC20350-1) was listed as 1.5 mg/kg on the reporting form and 0.0301 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0301 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00253 \text{ Kg} \times 78.9/100} = \frac{0.00301}{0.0019962} = 1.5079 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 1.5 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 1.5 mg/kg for Sample PPG174-MAIN-B11 was correctly reported. This was the highest detected Cr+6 concentration of the two detected results for the 2 soil samples of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Each of the two soil samples of this SDG were observed to fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentrations are also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total

chromium concentrations are all less than 14 mg/kg, thereby making it extremely unlikely that Cr+6 concentrations would increase to any significant degree.

Summary for Hexavalent Chromium Analysis – SDG JC20350

Since the soluble and insoluble MS spike recoveries were below QC limits in the QC sample of QC Batch GP97681, the soil sample in this QC batch required reanalysis. The remaining QC results associated with the hexavalent chromium analysis were within QC limits. Therefore, the Cr+6 results for samples JC20350-1 and JC20350-2 were qualified following the DV review and flagged with “NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix. Consequently, the soil samples of this QC Batch GP97681 were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analysis in JC20350”.

Cr+6 Re-analyses in SDG JC20350

Because the soluble and insoluble MS recoveries were below QC limits in the QC batch, the resultant data for the batch consisting of two soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC20350-1R and -2R in QC Batch GP98058, including the calibrations (r = 0.99993, 94.1 – 94.4% CCV Recoveries), QC blanks, duplicate analysis (≤ 2 × contract required quantitation limit [CRQL]), and blank spike analysis (90.5% – 96.0%). The soluble and insoluble MS recoveries were considerably improved in the reanalysis, the soluble increasing to 49.1% and the insoluble to a value within QC limits, as detailed below.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the soluble and insoluble MS recoveries in QC Sample JC20350-2R was considerably better than the initial analysis, but the soluble was still under the QC limits, while the insoluble MS recovery improved to a value within the QC limits of 75-125%, as observed below in Table 5. The post-digestion spike MS recovery was similar to the recovery value of the initial analysis, a value within QC limits of 85-115%.

Table 5. Hexavalent Chromium Re-analysis MS Recovery Results – JC20350

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98058 X	JC20350-2R	Cr ⁺⁶ , soluble	49.1 %	NJ-	Low
GP98058 X	JC20350-2R	Cr ⁺⁶ , insoluble	91.6 %	----	----
GP98058 X	JC20350-2R	Cr ⁺⁶ , post-digestion spike	87.6 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. X – The samples associated with QC Batch GP98058 consist of JC20350-1R and JC20350-2R.					

Since the soluble MS recovery in QC Batch GP98058 was still below the QC limits (75-125%), a recovery value marginally below 50%, the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” for a potential low bias in the

ability to recover Cr+6 in this QC batch. The qualified Cr+6 results of the reanalysis are presented below in Table 6, together with the results of the initial Cr+6 results.

The low soluble spike recovery seems to confirm a potential low bias in the ability to recover Cr+6 in this QC batch suggested by the initial analysis where the soluble MS recovery was 13.9%. The sample Cr+6 results of the two soil samples that were below the SCC value of 20 mg/kg in the initial analysis, remained below the SCC upon reanalysis, as well, though slightly lower, despite the vastly improved MS recoveries, differences possibly attributable to variability as the result of sample non-homogeneity.

Duplicate Analysis (QC Limit: $\leq 35\%$ RPD)

The duplicate analyses were performed on one set of duplicate soil sample aliquots. The difference between the duplicate soil sample aliquot concentrations for Cr+6 in the sample aliquots was listed as 200%RPD. Although this RPD value exceeded 35%, the analytical precision results were acceptable because of the low sample concentrations where the difference (0.38 mg/kg) between the raw concentration values was less than twice the reporting limit (2×0.47). Hence, the Cr+6 results in the associated samples were not qualified for the duplicate analysis result and analytical precision is considered acceptable in the re-analysis.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC20350-2RT (PPG174-MAIN-B12), a QC samples which was analyzed twice with a detected concentration of 1.2 mg/kg and a non-detect result in the re-analysis, values well below the SCC of 20 mg/kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. The total organic carbon (TOC) analysis was performed outside the 14-day analytical holding time. Hence, the TOC result is qualified as an estimated value and flagged with "J" in Table 6, below. In accordance with the method, these analyses were performed on the sample experiencing the low spike recoveries. A concentration of total organic carbon (298,000 mg/kg) and the ferrous iron (Fe+2) with a result of 0.79 % were detected in the QC sample in JC20350-2RT, thereby indicating the likely presence of a reducing soil matrix in both soil samples, as suggested by the presence of this soil sample below the Eh-pH phase line, as is the other soil sample of this SDG.

The "reducing" conditions in the soil matrix appear supported by the detected TOC concentration and the detected Fe⁺² data in support of the results of the Eh-pH analyses.

Summary for Hexavalent Chromium Analysis – SDGs JC20350

The qualified soil sample results from the initial Cr+6 analysis in SDG JC20350 are presented below in Table 6 alongside those qualified results obtained from the re-analysis of the samples. Both sets of analytical Cr+6 results for samples JC20350-1 and JC20350-2 and their re-analysis are still both qualified as estimated values (NJ-) due to a potential low bias, as the soluble MS recoveries were both below QC limits. The second analysis exhibited considerably improved MS recoveries, with the soluble MS recovery increasing to 49.1%, a value marginally below 50%, and the insoluble MS recovery improving to a value well within QC limits.

Table 6. Comparison of Qualified Cr⁺⁶ Results in JC20350 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC20350 Result (mg/kg)	DV Qualifier	JC20350-R Results (mg/kg)	DV Qualifier
PPG174-MAIN-B11	JC20350-1	Cr+6	1.5	NJ-	0.86	NJ-
PPG174-MAIN-B12	JC20350-2	Cr+6	1.2	NJ-	< 0.47	NJ-
PPG174-MAIN-B12	JC20350-2RT	TOC	----	----	298,000	J

mg/kg - milligrams per kilogram
 < –The analyte was analyzed for but was not detected above the stated reporting limit.
 J – The reported result is an estimated value.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

Although the soluble MS recovery in QC Batch GP97681 was less than 50% (13.9%) in the initial analysis with the results subject to rejection (NJDEP, 2009), the associated detected Cr+6 sample concentrations were only qualified as estimated values and flagged with “NJ-”, rather than be rejected, because inorganic data validation guidelines do not reject detected concentrations (US EPA, 2014), as well as other reasons considered as part of a **data usability** approach previously discussed with Mr. Joseph Sanguiliano of the NJDEP. The non-detect Cr+6 result for the reanalysis of JC20350-2 (JC20350-2R) was also not rejected, but only qualified as an estimated reporting limit (< 0.47 NJ-), because the soluble MS recovery in the reanalysis was just marginally below the 50% - 75% QC range where DV guidelines recommend qualification of associated results (NJDEP, 2009) and because the total chromium concentration of only 13.4 mg/kg is extremely unlikely to exceed the SCC of 20 mg/kg, especially since the soil the sample was collected from a “reducing” soil environment. This is further supported by the presence of both of these samples in a soil that displayed conditions of a “reducing” environment as indicated by the position of the samples below the Eh-pH phase diagram line with an extremely high concentration of TOC in JC20350-2RT, such that conversion to Cr+6 is not favored or anticipated.

The low MS recovery in the soluble matrix spike recovery suggests a potential low bias in the ability to recover Cr+6 in this QC batch. Consequently, professional judgement was applied in qualifying the Cr+6 results in both analyses as estimated values and flagged with “NJ-” due to a potential low bias, as suggested by the MS results tabulated above in Table 4 and Table 5. Furthermore, the concentrations obtained from the re-analysis within the 30-day holding time were slightly lower in the re-analysis, despite the much improved soluble and insoluble MS recoveries of the re-analysis supporting the decision not to reject the two detected Cr+6 results of the initial analysis associated with the poorer MS recoveries.

The TOC result in Sample JC20350-2RT is qualified as an estimated value and flagged with “J” in Table 6, above, because the analysis was performed outside the 14-day analytical holding time.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.

N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

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ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC20350/JC20350A

- 1. Were the appropriate sample preservation requirements met?..... Yes No
- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

The TOC result in Sample JC20350-2RT was qualified as an estimated value ("J") because the sample was analyzed 6 days past the 14-day holding time.

- 3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

- 5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

- 7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2, 4, and 5 for QC details. Qualified sample results are presented in Tables 3 and 6 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC20639/JC20639A
Sample Dates: May 19, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: June 25, 2016

This data validation (DV) report presents the data review and result qualifications for seven (7) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on May 19, 2016, for sample delivery group (SDG) JC20639, as well as JC20639A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC20639A and JC20639 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 7 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC20639-1A through JC20639-7A (inclusive)
- Chromium ("J") in Samples JC20639-6A and JC20639-7A
- Hexavalent chromium ("NJ-") in Samples JC20639-1 through JC20639-7 (inclusive)

No other sample results in SDG JC20639A and JC20639 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except the antimony results in Samples JC20639-1A and JC20639-4A, while the hexavalent chromium (Cr+6) concentrations were below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Tables 4 and 8 of this DV report.

Sample Receipt

The seven (7) post-excavation soil samples collected May 19, 2016, were received intact and preserved appropriately the same day, May 19, at the Accutest laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 3.5 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC20639A and JC20639

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-B13	JC20639-1A	5/19/2016	Soil	Metals
PPG174-MAIN-B14	JC20639-2A	5/19/2016	Soil	Metals
PPG174-MAIN-B15	JC20639-3A	5/19/2016	Soil	Metals
PPG174-MAIN-B16	JC20639-4A	5/19/2016	Soil	Metals
PPG174-MAIN-B17	JC20639-5A	5/19/2016	Soil	Metals
PPG174-MAIN-B17	JC20639-5AD	5/19/2016	Soil	Metals
PPG174-MAIN-B17	JC20639-5AS	5/19/2016	Soil	Metals
PPG174-MAIN-B18	JC20639-6A	5/19/2016	Soil	Metals
PPG174-DUP	JC20639-7A	5/19/2016	Soil	Metals
PPG174-MAIN-B13	JC20639-1	5/19/2016	Soil	Cr+6
PPG174-MAIN-B14	JC20639-2	5/19/2016	Soil	Cr+6
PPG174-MAIN-B15	JC20639-3	5/19/2016	Soil	Cr+6
PPG174-MAIN-B16	JC20639-4	5/19/2016	Soil	Cr+6
PPG174-MAIN-B17	JC20639-5	5/19/2016	Soil	Cr+6
PPG174-MSD	JC20639-5D	5/19/2016	Soil	Cr+6
PPG174-MS	JC20639-5S	5/19/2016	Soil	Cr+6
PPG174-MAIN-B18	JC20639-6	5/19/2016	Soil	Cr+6
PPG174-DUP	JC20639-7			
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids.				
Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JC20639A, while the data package for the hexavalent chromium analyses is numbered JC20639.

Data Review

Data, as presented in the analytical data packages SDG JC20639A and JC20639 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16* (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) *“National Functional Guidelines for Inorganic Data Review”*, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA *“ICP-AES Data Validation, SOP No. HW-2a, Revision 15”* (USEPA, 2012).

- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC20639A

The data validation of the metals analytical data in SDG JC20639A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|---------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | Field duplicate sample analysis |
| √ Data qualifiers | √ Data package completeness |

The 7 post-excavation soil samples were analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 7 samples of SDG JC20639A, the antimony results in 2 samples (JC20639-1A and JC20639-4A) exhibited a concentration above the IGWSSL of 6 milligrams per kilogram (mg/kg) for antimony. All remaining target metals results were less than the respective IGWSSL and SRS limits.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP93845 indicating possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution result for antimony was outside control limits; however, the percent difference (%D) result was acceptable due to a low initial sample antimony concentration (< 50 times instrument detection limit [IDL]). All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample and field blank analyses and acceptable analyte quantitation (concentration determination).

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC20639A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis
(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for PPG QC batch sample, JC20639-5A, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93845 Ω	JC20639-5A	Antimony	63.1 %	62.6 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate.						

NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ω – The samples associated with QC Batch MP93845 consist of JC20639-1A through -7A (inclusive).
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The antimony results in the seven affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 4.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 0.8 – 9.6%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 103.6% - 112.6% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative stated that the RPD serial dilution result for antimony was outside control limits in QC Batch MP93845, however, the percent difference (%D) result was acceptable due to a low initial sample concentration (< 50 times IDL). The remaining four serial dilution results associated with the soil samples ranged from 0 – 6.2%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Field Duplicate Sample Analysis (QC Limit ≤ 50 %RPD)

One set of field duplicate samples was collected as part of SDG JC20639A. Field duplicate sample collection and analysis can provide a determination of sampling representativeness and precision. Gross differences between field sample duplicates can be an indication of inconsistent sampling techniques or sample matrix complexities/non-homogeneity.

The advisory data validation guidelines for field duplicate soil sample analysis vary. There is no NJDEP DV guideline for qualifying field duplicate results (NJDEP, 2002). Recently, EPA has recommended qualifying field duplicate results that differ by more than 50%RPD or $> 2 \times$ contract required quantitation limit [CRQL] (US EPA, 2014), while the Field Sampling Plan for Hudson County chromium sites lists a data quality objective (DQO) of 50%RPD for soil samples (AECOM, 2010).

The results for the analysis of the one pair of field duplicate samples are presented in Table 3, below. It is apparent that the results for the metals analytes that were detected in the field duplicate samples were similar in the two field duplicate samples of PPG174-MAIN-B18, except for the total chromium results which differed by 145%RPD. Thus, the total chromium results of JC20639-6A and -7A are subject to qualification as estimated concentrations and are flagged with “J”, as the concentrations between field duplicate samples differed by more than 50%RPD. Soil sample non-homogeneity may be a contributor to the apparent disparity.

Table 3. Comparison of Field Duplicate Soil Sample Results – SDG JB20639A

Analyte	PPG174-MAIN-B18 (mg/kg)	PPG174-DUP (mg/kg)	% RPD	DV Flag
Antimony	2.6 NJ-	< 2.4 NJ-	< 2 × CRQL	-
Chromium	114	18.3	144.7	J
Nickel	25.5	24.8	2.8	-
Thallium	< 1.1	< 1.2	< 2 × CRQL	-
Vanadium	28.1	31.5	11.4	-
Total Solids	86.4 %	85.6 %	0.9 %	-
mg/kg - milligrams per kilogram < – The analyte was not detected at the stated reporting limit; J – The reported result is an estimated value; NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. CRQL – The value representing the US EPA Contract Laboratory Program contract required quantitation limit, often represented by the reporting limit, < 2 × CRQL – The difference between field duplicate results was less than two times the CRQL and meets QC requirements for sampling representativeness.				

Consequently, due to the disparity in the total chromium results for the field duplicate samples PPG174-MAIN-B18 and PPG174-DUP, these results are subject to qualification and are to be flagged with “J”, as indicated in Table 3 above and Table 4 below.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The post-excavation soil sample analytical results for the samples of SDG JC20639A were found to be compliant with the analytical methods for the analysis of metals in the 7 post-excavation soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP93845 associated with the seven soil samples: JC20639-1A through JC20639-7A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 4.

Table 4. Summary of Qualified Sample Metals Results in SDG JC20639A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-B13	JC20639-1A	Antimony	155	NJ-
PPG174-MAIN-B14	JC20639-2A	Antimony	< 2.1	NJ-
PPG174-MAIN-B15	JC20639-3A	Antimony	< 2.1	NJ-
PPG174-MAIN-B16	JC20639-4A	Antimony	9.5	NJ-
PPG174-MAIN-B17	JC20639-5A	Antimony	< 2.0	NJ-

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-B18	JC20639-6A	Antimony	2.6	NJ-
PPG174-MAIN-B18	JC20639-6A	Chromium	114	J
PPG174-DUP	JC20639-7A	Antimony	< 2.4	NJ-
PPG174-DUP	JC20639-7A	Chromium	18.3	J

Key:
mg/kg - milligrams per kilogram
< – The analyte was analyzed for, but was not detected above the stated reporting limit;
J – The reported result is an estimated value;
NJ- – The matrix spike recovery was below QC limits; associated sample result is an estimated value and may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC20639

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the seven post-excavation soil samples. The soil samples were re-analyzed in a second QC batch in SDG JC20639.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- √ Data qualifiers
- Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Field duplicate sample analysis

Hexavalent chromium was detected in each of the seven post-excavation soil samples analyzed in SDG JC20639, with sample Cr+6 results less than or equal to 8.2 mg/kg, values below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time, method blanks. However, the soluble matrix spike recovery in QC Batch GP97788 was outside control limits. The RPD value for the duplicate analysis in the analysis QC Batch GP97788 was above control limits, but the RPD was acceptable due to low duplicate and sample concentrations. No other QC requirements were exceeded.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99995 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 94.6% to 94.8% for the QC batch associated with the analysis of 7 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP97788 associated with the 7 soil samples of this SDG, as presented below in Table 5. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP97788 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. The remaining matrix spike recoveries were within the respective QC limits of 75-125% and 85-115%.

Table 5. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC20639

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97788 Ж	JC20639-5	Cr ⁺⁶ , soluble	71.6 %	NJ-	Low
GP97788 Ж	JC20639-5	Cr ⁺⁶ , insoluble	96.2 %	----	----
GP97788 Ж	JC20639-5	Cr ⁺⁶ , post-digestion spike	93.94 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
MS – Matrix spike
Cr⁺⁶ – Hexavalent chromium
NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.
Ж – The samples associated with QC Batch GP97788 consist of JC20639-1 through -7 (inclusive).

The Cr+6 results associated with a soluble MS recovery of 71.6% are qualified for the low spike recovery and are flagged with “NJ-”, in accordance with DV guidelines (NJDEP, 2009), which state that “if the spike recovery is ≥ 50% but ≤ 75% and the reported sample levels are greater than the method detection limit, flag the data as estimated “J” and indicate in the data validation report as to the potential low bias of the results.” These qualified Cr+6 results are presented together with the results from the re-analysis of this QC batch, which did not need qualification, below in Table 8.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from Sample location JC20639-5 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174_MAIN-B17) was 29.2%RPD, a value above the 20%RPD laboratory QC limit, but within the 35%RPD DV advisory QC limit for technical review of soil sample data (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (2.5%RPD) and pH (0.6%RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 88.5% and 83.2% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Field Duplicate Analysis (QC Limit ≤ 50%RPD)

The results for the analysis of one set of field duplicate samples are presented in Table 6, below. The difference for the low-level concentrations observed in the field duplicate samples from sampling location PPG174-MAIN-B18 differed by 42.2%RPD, which is below the QC limit of 50%RPD for soil samples (US EPA, 2014), as well as below the QC criterion of “≤ 2 × CRDL” for samples with analyte concentrations below 5 × CRDL.

Table 6. Comparison of Field Duplicate Soil Sample Results – SDG JC20639

Analyte	PPG174-MAIN-B18 (mg/kg)	PPG174-DUP (mg/kg)	% RPD	DV Flag
Hex.Chromium	0.91 NJ-	1.4 NJ-	42.4 %	----
mg/kg – milligrams per kilogram NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.				

Thus, the field duplicate results for the field duplicate samples from PPG174-MAIN-B18 demonstrated acceptable sampling representativeness and precision, with field duplicate soil sample results differing by less than 50%RPD. No soil sample Cr+6 results were qualified for sampling representativeness issues.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-MAIN-B14 (JC20639-2) was listed as 8.2 mg/kg on the reporting form and 0.1915 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.1915 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00256 \text{ Kg} \times 90.8/100} = \frac{0.01915}{0.0023245} = 8.2384 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 8.2 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 8.2 mg/kg for Sample PPG174-MAIN-B14 was correctly reported. This was the highest detected Cr+6 concentration of the seven detected results for the 7 soil samples of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Five soil samples were observed to fall below the Eh-pH phase diagram line, while two samples (JC20639-2 and -7) appeared to fall on or very near the phased line. The five samples that lie below the phase line represent samples that experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentrations are also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total chromium concentrations are all less than 86 mg/kg for the samples in the “reducing” soils and less than 19 mg/kg for the remaining two samples near the phase line. Hence, it seems highly unlikely that Cr+6 concentrations would increase to any significant degree, as observed in many other PPG data packages with total chromium concentrations below 600 mg/kg that exhibit Cr+6 results all (or almost all) concentrations are less than 20 mg/kg.

Hence, based on the sample total chromium and Cr+6 concentrations, it is highly unlikely that any of the affected samples including those in the “reducing” zone or the two samples near the “oxidizing” zone, would approach the SCC for Cr+6 of 20 mg/kg due to limitation created by the relatively low total chromium concentrations available for potential oxidation.

Summary for Hexavalent Chromium Analysis – SDG JC20639

Since the soluble MS spike recovery of 71.6% was below QC limits in the QC sample of QC Batch GP97788, the soil samples in this QC batch required reanalysis. The remaining QC results associated with the hexavalent chromium analysis were within QC limits. Therefore, the Cr+6 results for the seven samples of this QC batch in SDG JC20639 were qualified following the DV review and flagged with “NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix. Consequently, the soil samples of this QC batch were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analyses in SDG JC20639.”

Cr+6 Re-analyses in SDG JC20639

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the re-analysis batch consisting of 7 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC20639-1R through -7R in QC Batch GP98057, including the calibrations ($r = 0.99993$, 92.2 – 92.4% CCV Recoveries), QC blanks, duplicate analysis (9.6 %RPD), and blank spike analysis (95.0 and 93.3%), as well as all matrix spike recoveries. The soluble MS recovery was slightly higher in the reanalysis, improving to a

value within QC limits, while the post spike was also slightly better, meeting QC limits, as detailed below.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, all spike recoveries fell within the respective QC limits, as observed below in Table 7. The soluble MS recovery in JC20639-5R improved over the initial soluble MS recovery to exhibit an acceptable recovery.

Table 7. Hexavalent Chromium Re-analysis MS Recovery Results – JC20639

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98057 X	JC20639-5R	Cr ⁺⁶ , soluble	78.7 %	----	----
GP98057 X	JC20639-5R	Cr ⁺⁶ , insoluble	88.3 %	----	----
GP98057 X	JC20639-5R	Cr ⁺⁶ , post-digestion spike	95 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium X – The samples associated with QC Batch GP98057 consist of JC20639-1R through -7R (inclusive).					

Since the soluble MS recovery in QC Batch GP98057 improved to fall within the QC limits (75-125%), the Cr+6 results for the samples in this QC batch are not subject to qualification and are not to be flagged as estimated values. The “unqualified” Cr+6 results of the reanalysis are presented below in Table 8 together with the qualified (“NJ-”) results of the initial Cr+6 results.

Summary for Hexavalent Chromium Analysis – SDGs JC20639

The qualified soil sample results from the initial Cr+6 analysis in SDG JC20639 are presented below in Table 8 alongside those “un-qualified” results obtained from the reanalysis of the samples. The analytical Cr+6 results for samples JC20639-1 through -7 (inclusive) in the initial analysis are qualified as estimated values (NJ-) due to a potential low bias. The Cr+6 concentrations determined during the re-analysis of samples in SDG JC20639 analyzed within the 30-day holding time differ slightly from those of the initial analysis, but all are still well below the SCC of 20 mg/kg and are not subject to qualification due to the acceptability of all QC requirements of the re-analysis.

Table 8. Comparison of Qualified Cr⁺⁶ Results in JC20639 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC20639 Result (mg/kg)	DV Qualifier	JC20639-R Results (mg/kg)	DV Qualifier
PPG174-MAIN-B13	JC20639-1	Cr+6	0.49	NJ-	1.1	---
PPG174-MAIN-B14	JC20639-2	Cr+6	8.2	NJ-	0.47	---
PPG174-MAIN-B15	JC20639-3	Cr+6	0.85	NJ-	0.66	---
PPG174-MAIN-B16	JC20639-4	Cr+6	1.6	NJ-	1.7	---
PPG174-MAIN-B17	JC20639-5	Cr+6	0.73	NJ-	0.87	---
PPG174-MAIN-B18	JC20639-6	Cr+6	0.91	NJ-	1.3	---
PPG174-DUP	JC20639-7	Cr+6	1.4	NJ-	1.2	---
mg/kg – milligrams per kilogram < –The analyte was analyzed for but was not detected above the stated reporting limit. NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.						

Client ID	Laboratory Sample ID	Analyte	JC20639 Result (mg/kg)	DV Qualifier	JC20639-R Results (mg/kg)	DV Qualifier

Professional judgement was applied in qualifying the Cr+6 results in the initial analysis as estimated values (NJ-) due to a potential low bias, as suggested by the MS results tabulated above in Table 5, an approach consistent with DV guidelines (NJDEP, 2009). The Cr+6 results of the reanalysis did not warrant qualification as all QC requirements were achieved in the re-analysis.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, CLP, 2014, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9335.0-131, EPA-540-R-13-001, August 2014.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG **SDGs:** JC20639/JC20639A

1. Were the appropriate sample preservation requirements met?..... **Yes** No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? **Yes** No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes **No**
Indicate the identity of the samples and why.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes **No**
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? **Yes** No
If "Yes", include the number of samples and laboratory sample ID numbers.

The antimony results in Samples JC20639-1A and -4A exceeded the IGWSSL of 6 mg/kg.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... **Yes** No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... **Yes** No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No

9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**

If "yes", please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**

12. Were the QC Summary Forms reviewed?..... **Yes** No

13. Internal Standards acceptable..... **Yes** No

14. MS/MSD acceptable..... Yes **No**

15. Calibration summaries acceptable..... **Yes** No

16. Serial dilutions acceptable..... **Yes** No

17. Inorganic duplicates acceptable..... **Yes** No

18. LCS recovery acceptable..... **Yes** No

19. Other QC acceptable?..... Yes **No**
Provide a brief explanation, if applicable.

The total chromium results for field duplicate samples JC20639-6A and -7A differed by more than 50%RPD. These results for the field duplicate samples were qualified as estimated values and flagged with "J".

Refer to DV report tables 2, 3, 5, 6, and 7 for QC details. Qualified sample results are presented in Table 4 and Table 8 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC20878/JC20878A
Sample Dates: May 24, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: June 21, 2016

This data validation (DV) report presents the data review and result qualifications for two (2) post-excavation soil samples and one (1) field blank (FB) collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on May 24, 2016, for sample delivery group (SDG) JC20878, as well as JC20878A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample and field blank analytical results for the samples of SDG JC20878A and JC20878 were found to be compliant with the analytical methods employed for the analysis of vanadium and hexavalent chromium in the two collected post-excavation soil samples and one field blank.

Following the detailed DV review, no sample results required qualification.

Hence, no sample results in SDG JC20878A and JC20878 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals (vanadium) concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except the 98.1 milligrams per kilogram (mg/kg) vanadium result in JC20878-3, while the hexavalent chromium (Cr+6) concentration in the field blank was a non-detect result. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

Sample Receipt

The two soil samples and one field blank collected May 24, 2016, were received intact and appropriately preserved May 24 at the Accutest laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 4.4 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC20878A and JC20878

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-BERM-SW03R	JC20878-1	5/24/2016	Soil	Vanadium
PPG174-FB03	JC20878-2A	5/24/2016	Aqueous	Metals
PPG174-MAIN-SW03R	JC20878-3	5/24/2016	Soil	Vanadium
PPG174-FB03	JC20878-2	5/24/2016	Aqueous	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JC20878A, while the data package for the hexavalent chromium analyses is numbered JC20878.

Data Review

Data, as presented in the analytical data packages SDG JC20878A and JC20878 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of

data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC20878A

The data validation of the metals analytical data in SDG JC20878A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | √ Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 2 post-excavation soil samples were analyzed for only vanadium, while the field blank was analyzed for the 5 target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 2 soil samples of SDG JC20878A, the vanadium result in JC20878-3 exhibited a concentration above the SRS of 78 mg/kg for vanadium.

Laboratory Case Narrative

The case narrative basically stated that all QC requirements had been met for the metals analysis.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample and field blank analyses and acceptable analyte quantitation (concentration determination).

Consequently, no soil sample or field blank results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks, the continuing calibration blanks (CCBs) or the field blank at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC20878A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35% Relative Percent Difference [RPD])

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for vanadium were within the QC limits of 75 - 125% for non-client QC batch sample JC20336-2, as identified in Table 2 below.

Table 2. Matrix Spike Recovery Results

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP93769 Ω	JC20336-2	Vanadium	101.7 %	98.6 %	----	----
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias Ω – The samples associated with QC Batch MP93769 consist of JC20878-1 and JC20878-3						

The vanadium results in the two affected soil samples are not subject to qualification and the MS recovery results demonstrate acceptable accuracy.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples for only vanadium. The %RPD value was below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with a value of 1.8%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recovery of 100.5% for the vanadium result in the soil sample metals analysis, and 98.5 – 104.0% for the five target metals in the aqueous matrix.

Serial Dilution Analysis (QC Limit: ≤ 10 Percent Difference [%D])

The serial dilution result associated with the soil sample analysis for vanadium was an acceptable 7.4%D, a value below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC20878A were found to be compliant with the analytical methods for the analysis of metals in the two soil samples and one field blank using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, such that no soil sample or field blank target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC20878

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the one field blank.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was not detected in the one field blank of this SDG.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. The MS recovery for Cr+6 was outside control limits in a non-client sample indicating possible matrix interference. The case narrative stated that the 90% recovery in the pH-adjusted post spike was low. However, this recovery was within the 85-115% QC limits for a post spike analysis. There was good agreement between the samples and 1:5 dilution. No other QC requirements were exceeded.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99998 for the aqueous fraction, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 98.6 to 99.5% for the aqueous fraction, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg), the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]), or the field blank. Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery of 0% was below the QC limits of 75-125% for QC Batch GN46453 associated with the field blank of this SDG, as presented below in Table 3. However, the QC sample is from another client of unknown sample character.

Table 3. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC20878

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GN46453 ω	JC20679-11	Cr ⁺⁶ , soluble	0.0 %	NJ-	Low
GN46453 ω	JC20679-11	Cr ⁺⁶ , pH-adjusted post spike	90 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
MS – Matrix spike
Cr⁺⁶ – Hexavalent chromium
NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
ω – The sample associated with QC Batch GN46453 consists of JC20878-2.

Ordinarily, the Cr+6 result associated with this 0% MS recovery would be subject to qualification for the low spike recovery and either rejected or flagged with “NJ-” (NJDEP, 2009; USEPA, 2010). However, because the batch QC sample is from another client of unknown sample matrix character, qualification of the non-detect Cr+6 result in the field blank of this SDG is judged inappropriate. Many field blanks have been analyzed for the PPG project with no Cr+6 concentrations ever having been detected, regardless of the associated QC results. Because of the history of non-detect Cr+6 results in PPG field blanks and the acceptable pH-adjusted post spike recovery, professional judgement was applied in not qualifying the field blank result, based on the history of other PPG analyses involving field blank analysis and the unknown character of the non-client batch QC sample.

Duplicate Analysis (QC Limit: ≤ 20 %RPD)

The duplicate analysis was performed on one set of duplicate aqueous samples from a non-client sample (JC20679-11) for the aqueous sample fraction. The difference between the duplicate sample aliquots for Cr+6 in this sample was 0.0%RPD, a value below the 20%RPD laboratory QC limit (US EPA, 2010). Because the %RPD value for Cr+6 was below the QC limit, the associated sample results are acceptable and do not warrant qualification. Hence, the Cr+6 sample result in the field blank is not subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with a blank spike recovery of 100% for the aqueous matrix, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as serial dilution is not a requirement of the analytical method. The note in the case narrative indicated that there was good agreement between the sample and the 1:5 dilution in the QC batch.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the quantitation reports in the raw data. The following equation (NJDEP, 2009) was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/L)} = A \times E$$

Where: A = concentration from calibration curve (mg/L)
 E = Dilution (if necessary)

The non-detected hexavalent chromium concentration for Sample PPG174-FB03 (JC20878-2) was listed as < 0.010 mg/L on the reporting form and – 0.0028 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/L)} = A \times E$$

$$\text{Cr}^{+6} \text{ (mg/L)} = -0.0028 \text{ mg/L} \times 1 = -0.0028 \text{ mg/L}$$

$$\text{Cr}^{+6} \text{ (mg/L)} = -0.003 \text{ mg/L} = < 0.010 \text{ mg/L}$$

After rounding to three significant figures, this verifies that the non-detected hexavalent chromium concentration of < 0.010 mg/L for Sample PPG174-FB03 was correctly reported.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for the analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

The field blank sample was observed to fall below the Eh-pH phase diagram line, thereby suggesting that the sample experiences conditions of a “reducing” environment. The Cr+6 sample result in a reducing sample matrix is not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing conditions. The field blank sample Cr+6 concentration is a non-detect result and is also not expected to increase.

Summary for Hexavalent Chromium Analysis – SDG JC20878

Since the QC requirements were met in the field blank analysis, no Cr+6 results were subject to qualification

The reported sample results are usable without qualification, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
-----------	------------

J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC20878/JC20878A

- 1. Were the appropriate sample preservation requirements met?..... Yes No

- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

- 3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

- 5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The vanadium result in Sample JC20878-3 exceeded the SRS of 78 mg/kg.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

- 7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... Yes No

9. Were rejections noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes No

If "yes", please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds
of concern at the site met? Yes No

12. Were the QC Summary Forms reviewed?..... Yes No

13. Internal Standards acceptable..... Yes No

14. MS/MSD acceptable..... Yes No

15. Calibration summaries acceptable..... Yes No

16. Serial dilutions acceptable..... Yes No

17. Inorganic duplicates acceptable..... Yes No

18. LCS recovery acceptable..... Yes No

19. Other QC acceptable?..... Yes No
Provide a brief explanation, if applicable.

No sample results required qualification for the soil samples or field blank analysis.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC21195/JC21195A
Sample Dates: May 27, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: July 12, 2016

This data validation (DV) report presents the data review and result qualifications for four (4) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey from May 27, 2016, for sample delivery group (SDG) JC21195, as well as JC21195A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC21195A and JC21195 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 4 collected post-excavation soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC21195-2A through JC21195-5A (inclusive)
- Hexavalent chromium ("NJ-") in Samples JC21195-2 through JC21195-5 (inclusive)
- Hexavalent chromium ("NJ-") in reanalysis samples JC21195-2R through JC21195-5R (inclusive)

No other sample results in SDG JC21195A and JC21195 required qualification based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except the antimony result in Sample JC21195-2A, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in

Tables 3 and 6 of this DV report.

Sample Receipt

The four (4) soil samples collected May 27, 2016, were received intact and appropriately preserved the same day, May 27, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 5.4 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC21195A and JC21195

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-B20	JC21195-2A	5/27/2016	Soil	Metals
PPG174-MAIN-B21	JC21195-3A	5/27/2016	Soil	Metals
PPG174-MAIN-B13R	JC21195-4A	5/27/2016	Soil	Metals
PPG174-MAIN-B16R	JC21195-5A	5/27/2016	Soil	Metals
PPG174-MAIN-B20	JC21195-2	5/27/2016	Soil	Cr+6
PPG174-MAIN-B20	JC21195-2RT	5/27/2016	Soil	TOC, SS, Fe2+
PPG174-MAIN-B21	JC21195-3	5/27/2016	Soil	Cr+6
PPG174-MAIN-B13R	JC21195-4	5/27/2016	Soil	Cr+6
PPG174-MAIN-B16R	JC21195-5	5/27/2016	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting the metals data is numbered JC21195A, while the data package for the hexavalent chromium analyses is numbered JC21195. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC21195 together with the supplemental total organic carbon (TOC), sulfide screen, and ferrous iron. The samples data were validated for the five target metals (antimony, chromium, nickel, thallium, and vanadium), as were the hexavalent chromium data, and supplemental TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC21195A and JC21195 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.

- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC21195A

The data validation of the metals analytical data in SDG JC21195A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 4 post-excavation soil samples were analyzed for the 5 target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 4 soil samples of SDG JC21195A, only antimony in JC21195-2A exceeded the IGWSSL of 6 mg/kg; the remaining results were below the respective IGWSSL and SRS limits.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP94004 indicating

possible matrix interference and/or sample non-homogeneity. All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), except for thallium detected in CCB8 at 14:52 (2.1 micrograms per liter [µg/L]) and CCB 11 at 16:29 (2.5 µg/L) of analytical sequence MA39534. However, no soil sample results warranted qualification for any associated QC blank contamination in SDG JC21195A, because thallium was not detected in the associated samples and there is no positive bias in a non-detect result.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%Relative Percent Difference [RPD])

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for a non-client QC batch sample JC21032-2, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP94004 Ω	JC21032-2	Antimony	71.3 %	71.9 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ω – The samples associated with QC Batch MP94004 consist of JC21195-2A through -5A (inclusive).						

The antimony results in these four affected soil samples are flagged with “NJ-” due to a potential low bias. The metals concentrations in the non-client QC sample appear to be similar to those typically observed in PPG samples; therefore, qualification of the associated antimony results was judged appropriate in this case. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging from 1.9 - 4.3%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 94.4% - 100.0% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit ≤ 10 Percent Difference [%D])

The serial dilution results associated with the soil samples ranged from 0 – 9.3%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC21195A were found to be compliant with the analytical methods for the analysis of metals in the 4 soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP94004 associated with the 4 soil samples: JC21195-2A through JC21195-5A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JC21195A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-B20	JC21195-2A	Antimony	13.7	NJ-
PPG174-MAIN-B21	JC21195-3A	Antimony	< 2.2	NJ-
PPG174-MAIN-B13R	JC21195-4A	Antimony	< 2.3	NJ-
PPG174-MAIN-B16R	JC21195-5A	Antimony	3.6	NJ-
Key: mg/Kg – milligrams per kilogram				

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
<				-The analyte was analyzed for but was not detected above the stated reporting limit.
NJ-				-The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC21195

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the four post-excavation soil samples. The soil samples were re-analyzed in a second QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was detected in three of the four soil samples analyzed in SDG JC21195, with the sample Cr+6 result of 1.0 mg/kg being the highest concentration in the initial analysis. All values were below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike, post spike, and pH adjusted post spike recoveries in QC Batch GP97997 were outside control limits, as was the soluble MS recovery in the reanalysis QC Batch GP98098. The RPD value for the duplicate analysis in the analysis QC Batch GP97997 was above control limits due to possible sample non-homogeneity. There was not a good agreement between the sample and 1:5 dilution. The ferrous iron and sulfide screen test were analyzed after completion of Cr+6 testing (outside of normal hold time) in order to provide more information about the possible impact of the sample matrix on Cr+6 recoveries. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99997 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 96.6% to 97.1% for the QC batch associated with the analysis of 4 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery were below the QC limits of 75-125% for QC Batch GP97997 associated with the 4 soil samples of this SDG, as presented below in Table 4. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP97997 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. All remaining MS recoveries were within QC limits.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC21195

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP97997 ¥	JC21195-2	Cr ⁺⁶ , soluble	36.4 %	NJ-	Low
GP97997 ¥	JC21195-2	Cr ⁺⁶ , insoluble	89.3 %	----	----
GP97997 ¥	JC21195-2	Cr ⁺⁶ , post-digestion spike	64.84 %	NJ-	Low
GP97997 ¥	JC21195-2	Cr ⁺⁶ , pH-adjusted post spike	72.15 %	NJ-	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. ¥ – The samples associated with QC Batch GP97997 consist of JC21195-2 through -5 (inclusive).					

The Cr+6 results qualified for low spike recoveries are flagged with “NJ-” (NJDEP, 2009; US EPA, 2010), as tabulated below in Table 6, together with the qualified results from the re-analysis of this QC batch.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from sample JC21195-2. The difference between the duplicate soil sample aliquots for Cr+6 in soil this sample (PPG174-MAIN-B20) was reported as 200%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD QC limit for technical review of soil samples (US EPA, 2010; AECOM, 2010). A possible cause of the observed differences between the duplicate results may be attributable to sample non-homogeneity. However, due to the low sample concentrations, the QC limit becomes “≤ ± 2 × CRDL”, or two times the reporting limit. Consequently, since the difference between the non-detect result in JC21195-2 and the 0.84 mg/kg in the duplicate aliquot is less than two times the reporting limit (0.92 mg/kg), the duplicate result meets the QC limit and the sample results are not subject to qualification. The %RPD values for redox potential (0.5%RPD) and pH (1.7%RPD) displayed acceptable analytical precision results. Because the duplicate analysis for Cr+6 actually met QC limits, the associated sample results are not qualified following the DV review to indicate acceptable analytical precision.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 84.5% and 85.1% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as serial dilution is not a requirement of the analytical method and serial dilution is not addressed in DV guidelines (NJDEP, 2009). A 1:5 dilution was actually performed and there was poor agreement between the sample and the diluted aliquot. However, even though the results may be considered for qualification, the Cr+6 results are already qualified as estimated values and are flagged with "NJ-" for the low spike recoveries. No additional qualification is warranted.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)
 C = Wet weight of sample (kg)
 D = % Solids/100
 E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-MAIN-B13R (JC21195-4) was listed as 1.0 mg/kg (dry weight) on the reporting form and 0.0206 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0206 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00246 \text{ Kg} \times 83.0/100} = \frac{0.00206}{0.0020418} = 1.0089 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 1.0 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 1.0 mg/kg (dry weight) for Sample PPG174-MAIN-B13R was correctly reported. This was the highest detected Cr+6 concentration of the 3 detected results for the 4 soil samples of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

All four soil samples were observed to fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a "reducing" soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable

under the reducing soil conditions. The sample Cr+6 concentrations are also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total chromium concentrations are all less than 45 mg/kg, thereby making it highly unlikely that Cr+6 concentrations would increase to any significant degree.

Summary for Hexavalent Chromium Analysis – SDG JC21195

Since the soluble MS spike recovery of 36.4% was below QC limits in the QC samples of QC Batch GP97997, as well as below 50%, the soil samples in this QC batch required reanalysis. All remaining QC results associated with the hexavalent chromium analysis were within QC limits, except for the low post spike and pH-adjusted post spike recoveries. Therefore, the Cr+6 results for the four samples of this QC batch in SDG JC21195 were qualified following the DV review and flagged with “NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix. Consequently, the soil samples of this QC batch were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analyses in SDG JC21195.”

Cr+6 Re-analyses in SDG JC21195

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the re-analysis batch consisting of 4 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC21195-2R through -5R in QC Batch GP98098, including the calibrations (r = 0.99997, 98.4 – 98.7% CCV Recoveries), QC blanks, duplicate analysis (14.6%RPD), and blank spike analysis (92.8% and 86.0%). The soluble MS recovery was higher in the reanalysis, increasing to a recovery above 50%, but still below QC limits, while the post-digestion spike improved to a value within QC limits, as detailed below.

The reported Cr+6 results are reported in wet weight units for the re-analysis data. However, the usability of the data is not significantly affected, since the Cr+6 results are so low in concentration, being all less than or equal to 2.2 mg/kg (2.5 mg/kg dry weight) in the reanalysis. The DV reviewer did convert the wet weight results into dry weight results during the data review in order to better evaluate the data usability of the data.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the soluble MS recovery in QC Sample JC21195-2R was considerably better than the initial analysis improving to 58.7%, but still under the QC limits, while the insoluble MS recovery was similar to the initial recovery and still within the QC limits of 75-125%, as observed below in Table 5. The post-digestion spike MS recovery improved significantly in the re-analysis to fall within the 85-115% QC limits.

Table 5. Hexavalent Chromium Re-analysis MS Recovery Results – JC21195

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98098	JC21195-2R	Cr ⁺⁶ , soluble	58.7 %	NJ-	Low
GP98098	JC21195-2R	Cr ⁺⁶ , insoluble	86.9 %	----	----
GP98098	JC21195-2R	Cr ⁺⁶ , post-digestion spike	88.82 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
 MS – Matrix spike
 Cr⁺⁶ – Hexavalent chromium
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

⌘ – The samples associated with QC Batch GP98098 consist of JC21195-2R through -5R (inclusive).

Since the soluble MS recovery in QC Batch GP98098 improved to 58.7% but was still below the QC limits (75-125%), the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” (NJDEP, 2009) for a potential low bias in the ability to recover Cr+6 in this QC batch. The qualified Cr+6 results of the reanalysis are presented below in Table 6 together with the results of the initial Cr+6 results.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC21195-2RT (PPG174-MAIN-B20), a QC sample which was analyzed twice with a non-detect result initially and a detected concentration of 2.2 mg/kg reported for the re-analysis, values well below the SCC of 20 mg/kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. In accordance with the method, these analyses were performed on the sample experiencing the low spike recoveries. A concentration of total organic carbon (107,000 mg/kg wet weight; 121,000 mg/kg dry weight) and the ferrous iron (Fe+2) with a result of 0.35 % were detected in the QC sample JC21195-2RT, thereby indicating the likely presence of a reducing soil matrix in both samples, as suggested by the presence of this soil sample below the Eh-pH phase line, as are the other three soil samples of this SDG.

The “reducing” conditions in the soil matrix appear supported by the detected TOC concentration and the detected Fe⁺² data in support of the results of the Eh-pH analyses.

Summary for Hexavalent Chromium Analysis – SDGs JC21195

The qualified soil sample results from the initial Cr+6 analysis in SDG JC21195 are presented below in Table 6 alongside those qualified results obtained from the re-analysis of the samples. Both sets of analytical Cr+6 results for samples JC21195-2 through -5 and their re-analysis are still both qualified as estimated values (“NJ-”) due to a potential low bias, as the soluble MS recoveries were both below QC limits. The second analysis performed within the 30-day holding time exhibited improved MS recoveries, except the insoluble MS recovery, which was slightly lower, but still within QC limits.

The Cr+6 concentrations determined during the re-analysis of samples in SDG JC21195 differ slightly from those of the initial analysis, but all are still considerably below the SCC of 20 mg/kg. Although the soluble and post-digestion spike recoveries were significantly improved in the re-analysis, this improvement was not manifested in a marked increase in corresponding Cr+6 results, as the change in Cr+6 results were mixed, possibly due to sample non-homogeneity, rather than improved MS recoveries.

Table 6. Comparison of Qualified Cr+6 Results in JC21195 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC21195 Result (mg/kg)	DV Qualifier	JC21195-R Results (mg/kg)	DV Qualifier
PPG174-MAIN-B20	JC21195-2	Cr+6	< 0.46	NJ-	2.2	NJ-
PPG174-MAIN-B21	JC21195-3	Cr+6	0.55	NJ-	< 0.40	NJ-
PPG174-MAIN-B13R	JC21195-4	Cr+6	1.0	NJ-	0.94	NJ-
PPG174-MAIN-B16R	JC21195-5	Cr+6	0.89	NJ-	2.1	NJ-

Client ID	Laboratory Sample ID	Analyte	JC21195 Result (mg/kg)	DV Qualifier	JC21195-R Results (mg/kg)	DV Qualifier
mg/kg – milligrams per kilogram < –The analyte was analyzed for but was not detected above the stated reporting limit. NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.						

Although the Cr+6 results of the initial analysis may be subject to rejection because the soluble MS recovery of 36.4% was below the 50% criterion where DV guidelines recommend rejection of associated sample results (NJDEP, 2009), the Cr+6 results in both the initial and re-analysis were qualified as estimated values and not rejected due to a data usability approach and professional judgement based on the following considerations.

First of all, Cr+6 concentrations were detected in three of the four post-excavation samples, both in the initial analysis and the re-analysis, and inorganic data validation guidelines do not recommend rejection of detected results (US EPA, 2014). Because of the low Cr+6 results, the variability between the initial and re-analysis Cr+6 concentrations may be due more to sample non-homogeneity rather than correlated to the MS recoveries. Both analyses were performed within the 30-day analytical holding time. Because the 58.7% soluble MS recovery in the re-analysis falls between 50-75%, the associated sample Cr+6 results of the re-analysis are clearly to be qualified as estimated values (“flag the data as estimated with J”) based on current data validation guidance (NJDEP, 2009). In consideration of the extensive amount of Cr+6 analyses performed at various PPG sites, it appears that the ability to recover Cr+6 from PPG soil samples is correlated more with the insoluble MS recoveries than results of the soluble MS recoveries. Because the insoluble MS recoveries were both similar and within QC limits in the initial, as well as the re-analysis, this provides additional support for qualifying and not rejecting the Cr+6 results of the initial analysis.

These samples exhibited corresponding total chromium results less than 42 mg/kg, with three of the four samples containing less than 16 mg/kg chromium, making it highly improbable that sample Cr+6 concentrations would approach the SCC of 20 mg/kg for these samples experiencing reducing soil conditions.

Because of the acceptable insoluble MS recoveries, the soluble MS recovery in the re-analysis falling between 50-75% where DV guidelines recommend qualifying results with “J”, the very low total chromium concentrations (< 42 mg/kg) in the soil samples thereby limiting the degree to which chromium may be oxidized to Cr+6 in a “reducing” soil environment, and the similarity in detected Cr+6 results, it was judged appropriate to qualify the Cr+6 results as estimated values flagged with “NJ-” due to the possible low bias in the ability to recover Cr+6 from the “reducing” soil matrix. Rejection of Cr+6 results in the initial analysis for the low (36.4%) soluble MS recovery was judged not warranted based on the considerations noted above in a data usability perspective.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, CLP, 2014, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9335.0-131, EPA-540-R-13-001, August 2014.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC21195/JC21195A

- 1. Were the appropriate sample preservation requirements met?..... **Yes** No

- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? **Yes** No
If "No", provide a brief explanation.

- 3. Were the samples diluted? Yes **No**
Indicate the identity of the samples and why.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes **No**
If "Yes", list the affected samples.

- 5. Were any applicable standards exceeded for any samples? **Yes** No
If "Yes", include the number of samples and laboratory sample ID numbers.

The antimony result in JC21195-2A exceeded the IGWSSL of 6 mg/kg.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... **Yes** No
If "No", provide a brief explanation of action taken.

- 7. Were qualifications noted in the non-conformance summary?..... **Yes** No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.
- Not applicable**
10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2, 4, and 5 for QC details. Qualified sample results are presented in Tables 3 and 6 of this DV report.



DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC21302/JC21302A
Sample Dates: June 1, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: July 12, 2016

This data validation (DV) report presents the data review and result qualifications for nine (9) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on June 1, 2016, for sample delivery group (SDG) JC21302, as well as JC21302A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC21302A and JC21302 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 9 collected post-excavation soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC21302-1A through JC21302-9A (inclusive)
- Hexavalent chromium ("NJ-") in Samples JC21302-1 through JC21302-9 (inclusive)
- Hexavalent chromium ("NJ-") in reanalysis samples JC21302-1R through JC21302-9R (inclusive)

No other sample results in SDG JC21302A and JC21302 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except the nickel results in three samples: JC21302-1A, -3A, and -8A, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in

Tables 3 and 6 of this DV report.

Sample Receipt

The nine (9) soil samples collected June 1, 2016, were received intact and appropriately preserved the same day, June 1, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 4.5 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC21302A and JC21302

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-SW09	JC21302-1A	6/1/2016	Soil	Metals
PPG174-MAIN-SW10	JC21302-2A	6/1/2016	Soil	Metals
PPG174-MAIN-SW11	JC21302-3A	6/1/2016	Soil	Metals
PPG174-MAIN-SW12	JC21302-4A	6/1/2016	Soil	Metals
PPG174-MAIN-SW13	JC21302-5A	6/1/2016	Soil	Metals
PPG174-MAIN-SW14	JC21302-6A	6/1/2016	Soil	Metals
PPG174-MAIN-SW15	JC21302-7A	6/1/2016	Soil	Metals
PPG174-MAIN-SW16	JC21302-8A	6/1/2016	Soil	Metals
PPG174-MAIN-B22	JC21302-9A	6/1/2016	Soil	Metals
PPG174-MAIN-SW09	JC21302-1	6/1/2016	Soil	Cr+6
PPG174-MAIN-SW09	JC21302-1RT	6/1/2016	Soil	TOC, SS, Fe2+
PPG174-MAIN-SW10	JC21302-2	6/1/2016	Soil	Cr+6
PPG174-MAIN-SW11	JC21302-3	6/1/2016	Soil	Cr+6
PPG174-MAIN-SW12	JC21302-4	6/1/2016	Soil	Cr+6
PPG174-MAIN-SW13	JC21302-5	6/1/2016	Soil	Cr+6
PPG174-MAIN-SW14	JC21302-6	6/1/2016	Soil	Cr+6
PPG174-MAIN-SW15	JC21302-7	6/1/2016	Soil	Cr+6
PPG174-MAIN-SW16	JC21302-8	6/1/2016	Soil	Cr+6
PPG174-MAIN-B22	JC21302-9	6/1/2016	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting the metals data is numbered JC21302A, while the data package for the hexavalent chromium analyses is numbered JC21302. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC21302 together with the supplemental total organic carbon (TOC), sulfide screen and ferrous iron. The samples data were validated for the five target metals (antimony, chromium, nickel, thallium, and vanadium), as were the hexavalent chromium data, and supplemental TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC21302A and JC21302 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC21302A

The data validation of the metals analytical data in SDG JC21302A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 9 post-excavation soil samples were analyzed for the 5 target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 9 soil samples of SDG JC21302A, the nickel results in 3 samples (JC21302-1A, -3A, and -8A) were above the IGWSSL of 48 milligrams per kilogram (mg/kg). The remaining results were below the respective IGWSSL and SRS limits.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP94069 indicating possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution results for antimony and thallium were outside control limits in QC Batch MP94069; however, the percent difference (%D) results were acceptable due to the low initial sample concentrations (< 50 times instrument detection limit [IDL]). All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130%)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination). Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC21302A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for PPG QC batch sample JC21302-1A, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
----------	-----------	---------	-------------	--------------	--------------	----------------

MP94069 Ω	JC21302-1A	Antimony	59.0 %	54.5 %	NJ-	Low
QC Limits are 75-125% MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ω – The samples associated with QC Batch MP94069 consist of JC21302-1A through -9A (inclusive).						

The antimony results in the nine affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging from 4.3 – 14.5%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 92.9% - 97.0% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit ≤ 10 %D)

The case narrative stated that the RPD serial dilution results for antimony and thallium were outside control limits in QC Batch MP94069; however, the percent difference (%D) results were acceptable due to the low initial sample concentrations (< 50 times IDL). The remaining three serial dilution results associated with the soil samples ranged from 2.8 – 4.5%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC21302A were found to be compliant with the analytical methods for the analysis of metals in the 9 soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP94069 associated with the 9 soil samples: JC21302-1A through JC21302-9A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JC21302A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-SW09	JC21302-1A	Antimony	< 2.1	NJ-
PPG174-MAIN-SW10	JC21302-2A	Antimony	< 2.3	NJ-
PPG174-MAIN-SW11	JC21302-3A	Antimony	< 2.3	NJ-
PPG174-MAIN-SW12	JC21302-4A	Antimony	< 2.2	NJ-
PPG174-MAIN-SW13	JC21302-5A	Antimony	< 2.2	NJ-
PPG174-MAIN-SW14	JC21302-6A	Antimony	< 2.3	NJ-
PPG174-MAIN-SW15	JC21302-7A	Antimony	< 2.2	NJ-
PPG174-MAIN-SW16	JC21302-8A	Antimony	< 2.2	NJ-
PPG174-MAIN-B22	JC21302-9A	Antimony	2.4	NJ-

Key:
mg/kg – milligrams per kilogram
< –The analyte was analyzed for but was not detected above the stated reporting limit.
NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC21302

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the nine post-excavation soil samples. The soil samples were re-analyzed in a second QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was detected in each of the 9 soil samples analyzed in SDG JC21302, with all sample Cr+6 results less than 11 mg/kg, all values below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery in QC Batch GP98038 in the initial analysis was outside control limits, as were the soluble and insoluble matrix spike recoveries in re-analysis QC Batch GP98059 indicating possible matrix interference. Good post spike recoveries were achieved in both sets of analyses. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99994 for the initial soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 90.8% to 93.5% for the QC batch associated with the analysis of 9 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP98038 associated with the 9 soil samples of this SDG, as presented below in Table 4. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP98038 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. All remaining MS recoveries were within QC limits.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC21302

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98038 ¥	JC21302-1	Cr ⁺⁶ , soluble	55.6 %	NJ-	Low
GP98038 ¥	JC21302-1	Cr ⁺⁶ , insoluble	86.1 %	----	----
GP98038 ¥	JC21302-1	Cr ⁺⁶ , post-digestion spike	90.86 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
 MS – Matrix spike
 Cr⁺⁶ – Hexavalent chromium
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
 ¥ – The samples associated with QC Batch GP98038 consist of JC21302-1 through -9 (inclusive).

The Cr+6 results qualified for low spike recoveries are flagged with “NJ-” in accordance with DV guidelines, which recommend qualification of Cr+6 results as estimated values when associated with MS recoveries between 50 – 75% (NJDEP, 2009). These qualified Cr+6 results of the initial analysis are tabulated below in Table 6, together with the qualified results from the re-analysis of this QC batch.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from Sample JC21302-1 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-MAIN-SW09) was 16.2%RPD, a value below the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (0.1 %RPD) and pH (1.1 %RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 were below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 87.0% and 93.5% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (Kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-MAIN-SW16 (JC21302-8) was listed as 10.9 mg/kg on the reporting form and 0.2384 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.2384 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00250 \text{ Kg} \times 87.3/100} = \frac{0.02384}{0.0021825} = 10.9232 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 10.9 \text{ mg/kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 10.9 mg/kg for Sample PPG174-MAIN-SW16 was correctly reported. This was the highest detected Cr+6 concentration of the nine detected results for the 9 soil samples of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt (mV) solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. An exception consisted of Sample JC21302-1 falling above the Eh-pH phase diagram line and above the area of the graph because the Eh result of 707 mV was greater than the highest value of 700 mV on the Y-axis, hence representing a sample in the "oxidizing" area of the graph. No disparities relative to the

reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Seven of the nine seven soil samples appear to fall on or below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment, with four of these seemingly on the phase line. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. One sample was marginally above the line (JC21302-5) with JC21302-1 considerably above phase line and above the grid portion of the graph in the “oxidizing” zone. Hexavalent chromium was detected in each of the nine samples with corresponding total chromium concentrations ranging 7.3 to 347 mg/kg.

The ratios of total chromium to Cr+6 (Cr:Cr+6) in the samples of this SDG, irrespective of redox state of the soil, ranged from approximately 10 to 70, with those near the oxidizing portion of the phase diagram ranging 20 to 50. These ratios are not inconsistent with observations made in a Hudson County study where the average Cr+6 soil concentration at 42 sites consisting of residential and industrial sites was 2.6% of the average total chromium concentration, such that if a soil contained 1000 ppm of total chromium, then approximately 26 ppm was in the form of Cr+6 (Paustenbach, et al., 1991). Thus, the opportunity for the oxidation of total chromium to Cr+6 in the soil samples of this SDG appears to potentially be limited by the relatively low total chromium concentrations available.

Cr+6 Re-analyses in SDG JC21302

Because the soluble MS recovery was below QC limits in the initial analysis (QC Batch GP98038), the soil samples of this SDG required reanalysis. The resultant data for the batch consisting of 9 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC21302-1R through -9R in QC Batch GP98059, including the calibrations (r = 0.99979; 93.0 – 101.1% CCV Recoveries), QC blanks, duplicate analysis (15.4%RPD), and blank spike analysis (93.8% and 114.9%). The soluble MS recovery was almost identical to the initial analysis, while the insoluble MS recovery fell 30% to a value below QC limits in the reanalysis, but the post spike recovery was again acceptable in the re-analysis, as detailed below.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the soluble MS recovery in QC Sample JC21302-1R was similar to the initial analysis, but the insoluble MS recovery was now below the QC limits of 75-125%, as observed below in Table 5. The post-digestion spike MS recovery in JC21302-1R was still within the 85-115% QC limits.

Table 5. Hexavalent Chromium Re-analysis MS Recovery Results – JC21302

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98059 X	JC21302-1R	Cr ⁺⁶ , soluble	57.1 %	NJ-	Low
GP98059 X	JC21302-1R	Cr ⁺⁶ , insoluble	56.5 %	NJ-	Low
GP98059 X	JC21302-1R	Cr ⁺⁶ , post-digestion spike	92.2 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is					

estimated and may be biased low.

X – The samples associated with QC Batch GP98059 consist of JC21302-1R through -9R (inclusive).

Since the soluble MS and insoluble MS recoveries in QC Batch GP98059 fell within the range of 50-75% recovery, values below the QC limits of 75-125%, the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” for a potential low bias in the ability to recover Cr+6 in this QC batch (NJDEP, 2009). The qualified Cr+6 results of the reanalysis are presented below in Table 6 together with the results of the initial Cr+6 results.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC21302-1RT (PPG174-MAIN-SW09), a QC sample which was analyzed twice with detected concentrations of 4.0 and 3.0 mg/kg, values well below the SCC of 20 mg/kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. The total organic carbon (TOC) analysis was performed within the 14-day analytical holding time. In accordance with the method, these analyses were performed on the sample experiencing the low spike recoveries.

A concentration of total organic carbon (16,000 mg/kg) and the ferrous iron (Fe+2) with a result of 0.93 % were detected in the QC sample in JC21302-1RT, thereby indicating the likely presence of a “reducing” soil matrix in the soil sample. However, the presence of this soil sample above the Eh-pH phase line is in contrast to the indications of the TOC and ferrous iron results which strongly suggest a “reducing” soil environment with ample organic material to not foster oxidation of chromium. The presence of the TOC and ferrous iron in Sample JC21302-1 may be partly attributable as to why the Cr:Cr+6 ratio of 26 is present in the sample, similar to ratios observed for other PPG soil samples experiencing “reducing” soil conditions with corresponding Cr:Cr+6 ratios above 20.

Summary for Hexavalent Chromium Analysis – SDGs JC21302

The qualified soil sample results from the initial Cr+6 analysis in SDG JC21302 are presented below in Table 6 alongside those qualified results obtained from the re-analysis of the samples. Both sets of analytical Cr+6 results for samples JC21302-1 through -9 and their re-analysis are still both qualified as estimated values (NJ-) due to a potential low bias, as the soluble MS recoveries were both similar and below QC limits. Because the soluble MS recovery in the initial analysis, and the soluble and insoluble MS recoveries in the re-analysis fell between 50-75%, the Cr+6 results of both analyses are subject to qualification as estimated values to be flagged with “J” in accordance with DV guidelines (NJDEP, 2009) due to the potential low bias in the ability to recover the Cr+6 from the soil matrix.

The second analysis exhibited a lower recovery for the insoluble MS analysis in the re-analyses that were performed within the 30-day holding time, but a marked decrease in Cr+6 results was not evident. The Cr+6 results are still subject to qualification as estimated values in the re-analysis, because both the soluble and insoluble MS recoveries are below QC limits, but above 50% (NJDEP, 2009). The Cr+6 concentrations determined during the re-analysis of samples in SDG JC21302 differ slightly from those of the initial analysis, but all are still considerably below the SCC of 20 mg/kg.

Table 6. Comparison of Qualified Cr+6 Results in JC21302 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC21302 Result (mg/kg)	DV Qualifier	JC21302-R Results (mg/kg)	DV Qualifier
PPG174-MAIN-SW09	JC21302-1	Cr+6	4.0	NJ-	3.0	NJ-
PPG174-MAIN-SW10	JC21302-2	Cr+6	1.5	NJ-	2.3	NJ-
PPG174-MAIN-SW11	JC21302-3	Cr+6	9.4	NJ-	7.5	NJ-
PPG174-MAIN-SW12	JC21302-4	Cr+6	8.3	NJ-	4.7	NJ-
PPG174-MAIN-SW13	JC21302-5	Cr+6	10.6	NJ-	4.3	NJ-
PPG174-MAIN-SW14	JC21302-6	Cr+6	3.4	NJ-	1.2	NJ-
PPG174-MAIN-SW15	JC21302-7	Cr+6	2.6	NJ-	7.0	NJ-
PPG174-MAIN-SW16	JC21302-8	Cr+6	10.9	NJ-	8.0	NJ-
PPG174-MAIN-B22	JC21302-9	Cr+6	0.76	NJ-	0.95	NJ-
mg/kg – milligrams per kilogram NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.						

Professional judgement was applied in qualifying the Cr+6 results in both analyses as estimated values (NJ-) due to a potential low bias, as suggested by the MS results tabulated above in Table 4 and Table 5, an approach consistent with DV guidelines (NJDEP, 2009).

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

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New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

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New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

Paustenbach, D.J., Rinehart, W.E., and P.J. Sheehan, 1991, "The Health Hazards Posed by Chromium-Contaminated Soils in Residential and Industrial Areas: Conclusions of an Expert Panel" in ***Regulatory Toxicology and Pharmacology***, 13, pp 195-222 (1991).

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US EPA, CLP, 2010, ***"National Functional Guidelines for Inorganic Superfund Data Review"***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC21302/JC21302A

- 1. Were the appropriate sample preservation requirements met?..... Yes No

- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

- 3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

- 5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The nickel results in JC21302-1A, -3A, and -8A exceeded the IGWSSL of 48 mg/kg.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

- 7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.
- Not applicable**
10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2, 4, and 5 for QC details. Qualified sample results are presented in Tables 3 and 6 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC21391/JC21391A
Sample Dates: June 2, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: July 14, 2016

This data validation (DV) report presents the data review and result qualifications for three (3) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on June 2, 2016, for sample delivery group (SDG) JC21391, as well as JC21391A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC21391A and JC21391 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 3 collected post-excavation soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC21391-1A through JC21391-3A (inclusive)
- Hexavalent chromium ("*NJ-") in Samples JC21391-1 through JC21391-3 (inclusive)
- Hexavalent chromium ("NJ-") in reanalysis samples JC21391-1R through JC21391-3R (inclusive)

No other sample results in SDG JC21391A and JC21391 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) concentrations were also all below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Tables 3 and 7 of this DV report.

Sample Receipt

The three (3) soil samples collected June 2, 2016, were received intact and appropriately preserved the same day, June 2, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 5.7 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC21391A and JC21391

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-B23	JC21391-1A	6/2/2016	Soil	Metals
PPG174-MAIN-B24	JC21391-2A	6/2/2016	Soil	Metals
PPG174-MAIN-B25	JC21391-3A	6/2/2016	Soil	Metals
PPG174-MAIN-B23	JC21391-1	6/2/2016	Soil	Cr+6
PPG174-MAIN-B23	JC21391-1RT	6/2/2016	Soil	TOC, SS, Fe2+
PPG174-MAIN-B24	JC21391-2	6/2/2016	Soil	Cr+6
PPG174-MAIN-B25	JC21391-3	6/2/2016	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting the metals data is numbered JC21391A, while the data package for the hexavalent chromium analyses is numbered JC21391. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC21391 together with the supplemental total organic carbon (TOC), sulfide screen and ferrous iron. The samples data were validated for the five target metals (antimony, chromium, nickel, thallium, and vanadium), as were the hexavalent chromium data, and supplemental TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC21391A and JC21391 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.

- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC21391A

The data validation of the metals analytical data in SDG JC21391A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 3 post-excavation soil samples were analyzed for the 5 target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 3 soil samples of SDG JC21391A, none of the detected metals results were above the respective IGWSSL or SRS.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP94069 indicating possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution results for antimony and thallium were outside control limits in QC Batch MP94069; however, the percent difference (%D) results were acceptable due to the low initial sample antimony and thallium concentrations (< 50 times

instrument detection limit [IDL]). All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

The exceptions consisted of the 167.7% recovery of antimony in CRID1 at 10:08 and 143.3% in CRID2 at 13:36 in analytical sequence MA39557 associated with the 3 soil samples. However, the soil sample results were not affected because the reporting limits for antimony are above the respective affected range where results may be subject to qualification. The affected range is approximately 0 – 0.75 milligrams per kilogram (mg/kg) for antimony where the corresponding reporting limits are approximately 2 mg/kg, while the detected result in JC21391-3A was 5 mg/kg, all being above the affected range, such that qualification of results is not applicable.

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC21391A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for PPG QC batch sample JC21302-1A, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP94069 Ω	JC21302-1A	Antimony	59.0 %	54.5 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ω – The samples associated with QC Batch MP94069 consist of JC21391-1A through -3A (inclusive).						

The antimony results in the three affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging from 4.3 – 14.5%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 95.0% - 101.2% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit ≤ 10 %D)

The case narrative stated that the RPD serial dilution results for antimony and thallium were outside control limits in QC Batch MP94069; however, the percent difference (%D) results were acceptable due to a low initial sample concentrations (< 50 times IDL). The remaining three serial dilution results associated with the soil samples ranged from 2.8 – 4.5%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC21391A were found to be compliant with the analytical methods for the analysis of metals in the 3 soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP94069 associated with the 3 soil samples: JC21391-1A through JC21391-3A (inclusive). The antimony results in these samples are qualified as estimated

values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JC21391A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-B23	JC21391-1A	Antimony	< 2.2	NJ-
PPG174-MAIN-B24	JC21391-2A	Antimony	< 2.3	NJ-
PPG174-MAIN-B25	JC21391-3A	Antimony	5.0	NJ-

Key:
 mg/kg – milligrams per kilogram
 < –The analyte was analyzed for but was not detected above the stated reporting limit.
 NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC21391

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the three post-excavation soil samples. The soil samples were re-analyzed in a second QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- Matrix spike recoveries
- Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Data qualifiers

Hexavalent chromium was detected in one of the 3 soil samples analyzed in SDG JC21391, with the sample Cr+6 result of 7.2 mg/kg, all values below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery in QC Batch GP98054 was outside control limits, as was the soluble matrix spike recovery in reanalysis QC Batch GP98112. The RPD values for the duplicate analysis in the initial analysis QC Batch GP98054 and re-analysis QC Batch GP98112 were above control limits due to possible sample non-homogeneity. However, the RPD value for the duplicate analysis in the re-analysis QC batch was less than 35%RPD. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99979 for the initial soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 93.8% to 94.2% for the

QC batch associated with the analysis of 3 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP98054 associated with the 3 soil samples of this SDG, as presented below in Table 4. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP98054 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. All remaining MS recoveries were within QC limits.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC21391

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98054 ¥	JC21391-1	Cr ⁺⁶ , soluble	48.0 %	NJ-	Low
GP98054 ¥	JC21391-1	Cr ⁺⁶ , insoluble	97.2 %	----	----
GP98054 ¥	JC21391-1	Cr ⁺⁶ , post-digestion spike	90.92 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. ¥ – The samples associated with QC Batch GP98054 consist of JC21391-1 through -3 (inclusive).					

The Cr+6 results qualified for low spike recoveries are flagged with “NJ-” (US EPA, 2010), as tabulated below in Table 7, together with the qualified results from the re-analysis of this QC batch.

Although the soluble MS recovery in QC Batch GP98054 was only slightly less than 50% (48.0%) where the results may be subject to rejection (NJDEP, 2009), the associated detected Cr+6 sample concentration was only qualified as an estimated value and flagged with “NJ-“, rather than be rejected, because DV guidelines do not recommend rejection of detected concentrations (US EPA, 2014), and the insoluble recovery (97.2%) was within QC limits, a **data usability** approach discussed with Mr. Joseph Sanguiliano of the NJDEP. The non-detect Cr+6 results for JC21391-2 and -3 were also not rejected and only qualified as an estimated reporting limit (< 0.47 NJ- and < 0.45 NJ-) because the total chromium concentrations were both below 30 mg/kg for soils in a “reducing” soil environment and are unlikely to exceed the SCC of 20 mg/kg, because the sample was collected from a “reducing” soil environment with a limiting total chromium concentration. This is supported by the presence of these samples in a soil that displayed conditions of a “reducing” environment where oxidation to Cr+6 is not anticipated. The low MS recovery in the soluble matrix spike recovery suggests a potential low bias in the ability to recover Cr+6 in this QC batch. Consequently, the soil samples of this QC batch are qualified as estimated values and flagged with “*NJ-” in the re-analysis data set, as represented in Table 7.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from sample JC21391-1. The difference between the duplicate soil sample aliquots for Cr+6 in soil this sample

(PPG174-MAIN-B23) was 54.9%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD QC limit for technical review of soil samples (US EPA, 2010; AECOM, 2010), as presented below in Table 5. A possible cause of the observed differences between the duplicate results may be attributable to sample non-homogeneity. The %RPD values for redox potential (5.3%RPD) and pH (2.5%RPD) displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was above the QC limit for soil samples, the associated sample results are qualified as estimated values with an indeterminate bias and are flagged with “*J” to indicate that there is potential variability in the analytical precision.

Table 5. Duplicate Analysis Results Outside QC Limits

QC Batch	QC Sample	Analyte	Original Result (mg/kg)	Duplicate (mg/kg)	Difference	DV Qualifier
GP98054 ¥	JC21391-1	Cr+6	7.2	4.1	54.9 %RPD	*J
mg/kg – milligrams per kilogram QC Limit is 35%RPD * – Duplicate analysis not within control limits; indeterminate bias direction. J – The reported result is an estimated value. ¥ – The samples associated with QC Batch GP98054 consist of JC21391-1, -2, and -3.						

Since the duplicate analysis for Cr+6 had exceeded the QC limit for duplicate soil sample analysis, the associated PPG samples with laboratory ID numbers JC21391-1 through -3 (inclusive) were qualified as estimated values due to the potential variability in the analytical precision. Because the soluble MS recovery was also below QC limits, the Cr+6 results for these three soil samples are flagged with “*NJ-” in the summary table below, Table 7.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 91.8% and 98.8% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)
 C = Wet weight of sample (kg)

D = % Solids/100
E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-MAIN-B23 (JC21391-1) was listed as 7.2 mg/kg on the reporting form and 0.1562 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.1562 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00244 \text{ Kg} \times 88.7/100} = \frac{0.01562}{0.0021643} = 7.2172 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 7.2 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 7.2 mg/kg for Sample PPG174-MAIN-B23 was correctly reported. This was the only detected Cr+6 concentration of the three soil samples of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Each of the three soil samples was observed to fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentrations are also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total chromium concentrations are all less than 43 mg/kg, thereby making unlikely that Cr+6 concentrations would increase to any significant degree and approach the SCC of 20 mg/kg.

Summary for Hexavalent Chromium Analysis – SDG JC21391

Since the soluble MS spike recovery of 48% was below QC limits in the QC sample of QC Batch GP98054, the soil sample in this QC batch required reanalysis. The Cr+6 results for the associated samples were qualified following the DV review and flagged with “*NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix, as well as potential variability in the analytical precision as suggested by the duplicate analysis result. Consequently, the soil samples of this QC Batch GP98054 were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analysis in JC21391.”

Cr+6 Re-analysis in SDG JC21391

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the re-analysis of the batch consisting of 3 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC21391-1R through -3R in QC Batch

GP98112, including the calibrations ($r = 0.99990$, 98.2 – 101.1% CCV Recoveries), QC blanks, duplicate analysis (23.2%RPD < 35%RPD QC limit), and blank spike analysis (99.5% – 105.7%). The soluble MS recovery was considerably higher in the re-analysis, but still below QC limits, while the insoluble MS and post spike recoveries were also slightly higher and within the respective QC limits, as detailed below.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the re-analysis of the affected samples. However, upon reanalysis, the soluble MS recovery in QC Sample JC21391-1R was considerably better than the initial analysis, but still under the QC limits, while the insoluble MS recovery was improved and still within the QC limits of 75-125%, as observed below in Table 6. The post-digestion spike MS recovery in JC21391-1R was still within the 85-115% QC limits.

Table 6. Hexavalent Chromium Re-analysis MS Recovery Results – JC21391

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98112 X	JC21391-1R	Cr ⁺⁶ , soluble	67.9 %	NJ-	Low
GP98112 X	JC21391-1R	Cr ⁺⁶ , insoluble	113.0 %	----	----
GP98112 X	JC21391-1R	Cr ⁺⁶ , post-digestion spike	101.37 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. X – The samples associated with QC Batch GP98112 consist of JC21391-1R through -3R (inclusive).					

Since the soluble MS recovery in QC Batch GP98112 was still below the QC limits (75-125%), but above 50%, the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” for a potential low bias in the ability to recover Cr+6 in this QC batch, in accordance with DV guidelines (NJDEP, 2009) which recommend qualifying sample results associated with MS recoveries between 50-75% by flagging the results with “J.” The qualified Cr+6 results of the reanalysis are presented below in Table 7 together with the results of the initial Cr+6 results.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis in the re-analysis was performed on one set of duplicate soil sample aliquots. The difference between the duplicate soil sample aliquot concentrations for Cr+6 in the sample aliquots was listed as 23.3%RPD. Since this RPD value was below the 35%RPD criterion where DV guidelines recommend qualification of soil sample results (US EPA, 2014), the analytical precision results were acceptable, because the observed difference was less than 35%RPD. Hence, the Cr+6 results in the associated samples were not qualified for the duplicate analysis result and analytical precision is considered acceptable in the re-analysis.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC21391-1RT (PPG174-MAIN-B23), a QC sample which was analyzed twice with detected Cr+6 concentrations of 7.2 and 4.8 mg/kg, values below the SCC of 20 mg/kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional judgement was

applied in not qualifying the affected sulfide screen and ferrous iron data. The total organic carbon (TOC) analysis was performed inside the 14-day analytical holding time. In accordance with the method, these analyses were performed on the sample experiencing the low spike recoveries. A concentration of total organic carbon (15,900 mg/kg) and the ferrous iron (Fe+2) with a result of 0.22 % were detected in the QC sample in JC21391-1RT, thereby indicating the likely presence of a reducing soil matrix in the soil sample, as suggested by the presence of this soil sample below the Eh-pH phase line, as are the other two soil samples of this SDG.

The “reducing” conditions in the soil matrix appear supported by the detected TOC concentration and the detected Fe⁺² data in support of the results of the Eh-pH analyses.

Summary for Hexavalent Chromium Analysis – SDGs JC21391

The qualified soil sample results from the initial Cr+6 analysis in SDG JC21391 are presented below in Table 7 alongside those qualified results obtained from the re-analysis of the samples. Both sets of analytical Cr+6 results for samples JC21391-1 through -3 and their re-analysis are still both qualified as estimated values (NJ-) due to a potential low bias, as the soluble MS recoveries were both below QC limits of 75-125%. The second analysis exhibited higher recoveries for the soluble MS, insoluble MS and post spike recoveries in the re-analysis that was performed within the 30-day holding time. The Cr+6 results are still subject to qualification as estimated values in the re-analysis, because the soluble MS recovery recovered below QC limits, but within 50-75% where DV guidelines (NJDEP, 2009) recommend qualification of associated Cr+6 results as estimated values to be flagged with “J.” The results of the initial analysis are also qualified for the observed variability in the duplicate analysis such that the results of the initial analysis are qualified as estimated values and flagged with “*NJ-” due to a potential low bias in the ability to recover Cr+6 from the “reducing” soil matrix and possible variability in the analytical precision.

The Cr+6 concentrations determined during the re-analysis of samples in SDG JC21391 differ slightly in only one sample from that of the initial analysis for JC21391-1. The non-detect results for the other two samples, despite the improved soluble, insoluble and post-digestion spike recoveries in the re-analysis are still non-detect values. The results of the three samples are still considerably below the SCC of 20 mg/kg.

Table 7. Comparison of Qualified Cr+6 Results in JC21391 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC21391 Result (mg/kg)	DV Qualifier	JC21391-R Results (mg/kg)	DV Qualifier
PPG174-MAIN-B23	JC21391-1	Cr+6	7.2	*NJ-	4.8	NJ-
PPG174-MAIN-B24	JC21391-2	Cr+6	< 0.47	*NJ-	< 0.47	NJ-
PPG174-MAIN-B25	JC21391-3	Cr+6	< 0.45	*NJ-	< 0.45	NJ-

mg/kg – milligrams per kilogram
 < – The analyte was analyzed for but was not detected above the stated reporting limit.
 * – Duplicate analysis not within control limits; indeterminate bias direction.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

Professional judgement was applied in qualifying the Cr+6 results in both analyses as estimated values (NJ-) due to a potential low bias, as suggested by the soluble MS results tabulated above in Table 4 and Table 6.

While the soluble MS recovery of 48% was just slightly below the 50% criterion below which NJDEP DV guidelines recommend rejection of associated results, the Cr+6 results in the three

samples of SDG JC21391 were qualified as estimated values using professional judgement based on data usability concepts. The soluble MS recovery in the re-analysis (67.9%) was improved and fell well within the QC range of 50-75% where NJDEP DV guidelines recommend qualification of associated (detected and non-detected) results as estimated values to be flagged with "J." Because the non-detect results in samples JC21391-2 and -3 were confirmed in the re-analysis, despite the increase in the soluble MS, insoluble MS, and post spike recoveries, and the Cr+6 result in JC21391-1R was almost half of that detected in the initial analysis of JC321391-1, despite the increase in each of the spike recoveries, it was judged inappropriate to reject the results of the initial analysis. The detected Cr+6 results in JC21391-1 and JC21391-1R were qualified and not considered for rejection, because inorganic DV guidelines do not recommend rejection of detected concentrations associated with low matrix spike recoveries, even those <30% (US EPA, 2014). Additionally, based on the many soil samples analyzed for Cr+6 at the various PPG sites, it appears that the insoluble MS recovery results may be better indicators of the ability of the analysis to recover Cr+6 from the given soil matrix.

The results of Cr+6 analysis in samples of SDG JC21391 exhibited reasonably similar results in each of the three samples that were re-analyzed within the 30-day holding time, all Cr+6 results considerably below the SCC of 20 mg/kg.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
*	Duplicate analysis not within control limits; indeterminate bias direction.
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, CLP, 2014, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9335.0-131, EPA-540-R-13-001, August 2014.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC21391/JC21391A

- 1. Were the appropriate sample preservation requirements met?..... **Yes** No
- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? **Yes** No
If "No", provide a brief explanation.
- 3. Were the samples diluted? Yes **No**
Indicate the identity of the samples and why.
- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes **No**
If "Yes", list the affected samples.
- 5. Were any applicable standards exceeded for any samples? Yes **No**
If "Yes", include the number of samples and laboratory sample ID numbers.
- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... **Yes** No
If "No", provide a brief explanation of action taken.
- 7. Were qualifications noted in the non-conformance summary?..... **Yes** No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No

9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**

If “yes”, please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds
of concern at the site met? Yes **No**

12. Were the QC Summary Forms reviewed?..... **Yes** No

13. Internal Standards acceptable..... **Yes** No

14. MS/MSD acceptable..... Yes **No**

15. Calibration summaries acceptable..... **Yes** No

16. Serial dilutions acceptable..... **Yes** No

17. Inorganic duplicates acceptable..... Yes **No**

18. LCS recovery acceptable..... **Yes** No

19. Other QC acceptable?..... **Yes** No

Provide a brief explanation, if applicable.

Refer to DV report Tables 2, 4, 5, and 6 for QC details. Qualified sample results are presented in Tables 3 and 7 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC21496/JC21496A
Sample Dates: June 3, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: June 27, 2016

This data validation (DV) report presents the data review and result qualifications for two (2) post-excavation soil samples and one (1) field blank (FB) collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on June 3, 2016, for sample delivery group (SDG) JC21496, as well as JC21496A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample and field blank analytical results for the samples of SDG JC21496A and JC21496 were found to be compliant with the analytical methods employed for the analysis of vanadium, antimony and hexavalent chromium in the two collected post-excavation soil samples and one field blank.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Sample JC21496-3

No other sample results in SDG JC21496A and JC21496 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. The hexavalent chromium result in the field blank was not qualified following the DV review, because all QC results were within method QC limits. Details are provided in the tables and text below.

The reported metals (vanadium) concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) concentration in the field blank was a non-detect result, below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Table 3 of this DV report.

Sample Receipt

The two soil samples and one field blank collected June 3, 2016, were received intact and appropriately preserved June 3 at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 4.9 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC21496A and JC21496

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-FB04	JC21496-1A	6/3/2016	Aqueous	Metals
PPG174-MAIN-SW03R2	JC21496-2	6/3/2016	Soil	Vanadium
PPG174-MAIN-B20R	JC21496-3	6/3/2016	Soil	Antimony
PPG174-FB04	JC21496-1	6/3/2016	Aqueous	Cr+6

Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids, as appropriate.
Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.

The data package presenting the metals data is numbered JC21496A, while the data package for the hexavalent chromium analysis is numbered JC21496.

Data Review

Data, as presented in the analytical data packages SDG JC21496A and JC21496 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced”

deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC21496A

The data validation of the metals analytical data in SDG JC21496A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 2 post-excavation soil samples were analyzed for only vanadium and antimony, while the field blank was analyzed for the 5 target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 2 soil samples of SDG JC21496A, none were above the SRS limit of 78 milligrams per kilogram (mg/kg) for vanadium or the IGWSSL limit of 6 mg/kg for antimony.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP94069 indicating possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution result for chromium was outside control limits in QC Batch MP94104 associated with the field blank, while the RPD serial dilution result for antimony was outside control limits in QC Batch MP94069 associated with the soil samples; however, the percent difference (%D) results were acceptable due to the low initial sample concentrations (< 50 times instrument detection limit [IDL]). All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample and field blank analyses and acceptable analyte quantitation (concentration determination).

Consequently, no soil sample or field blank results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks, the continuing calibration blanks (CCBs) or the field blank at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC21496A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis
(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for PPG QC batch sample JC21302-1A from another SDG, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits, including those of QC Batch GP94104 associated with the field blank.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP94069 Ω	JC21302-1A	Antimony	59.0 %	54.5 %	NJ-	Low

QC Limits are 75-125%
MS – Matrix spike
MSD – Matrix spike duplicate.
NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.
Ω – The samples associated with QC Batch MP94069 consist of JC21496-2 and JC21496-3.

The non-detect antimony result in the affected soil sample (JC214896-3) is flagged with “NJ-” due to a potential low bias. The qualified antimony result is presented below in the summary table, Table 3.

Duplicate analysis (QC Limits: aqueous ≤ 20 %RPD; soils ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging from 4.7 – 9.8%RPD for soil samples and 3.1 – 3.8 %RPD for the batch QC sample associated with the field blank analysis with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 105.6% - 109.7% for the soil sample metals analysis, and 95.5 – 98.0% for the aqueous matrix.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative stated that the RPD serial dilution result for antimony was outside control limits in QC Batch MP94069 associated with the soil samples, while chromium was outside control limits in QC batch MP94104 associated with the field blank. However, the percent difference (%D) results were acceptable due to the low initial sample concentrations (< 50 times IDL). No sample or field blank results were qualified for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The sample analytical results for the samples of SDG JC21496A were found to be compliant with the analytical methods for the analysis of metals in the two soil samples and one field blank using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP94069 associated with the 2 soil samples. The antimony result in JC21496-3 is qualified as an estimated value (flagged “NJ-”) in the associated soil sample due to a potential low bias, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JC21496A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-B20R	JC21496-3	Antimony	< 2.5	NJ-
Key: mg/kg – milligrams per kilogram < – The analyte was analyzed for, but was not detected above the stated reporting limit NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.				

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC21496

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The field blank sample was analyzed in one QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- √ Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Data qualifiers

Hexavalent chromium was not detected in the field blank analyzed in SDG JC21496.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the method blank, as well as matrix spike recoveries. The sample was received out of holding time for pH, since this is considered a field analysis, and was analyzed by request. No QC requirements were exceeded.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99990 for the aqueous fraction, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 101.1% to 101.6% for the QC batch associated with the analysis of the aqueous fraction, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg), the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]), or the field blank. Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was within the QC limits of 75-125% for QC Batch GN46984 associated with the field blank of this SDG, as presented below in Table 4. Thus, the hexavalent chromium result in the field blank associated with this non-client QC batch sample did not warrant qualification based on the acceptable result of the soluble MS recovery.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC21496

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GN46984 ω	JC21504-2	Cr ⁺⁶ , soluble	90.0 %	----	----
QC Limits are 85-115% for MS recovery					

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium ω – The sample associated with QC Batch GN46984 consists of JC21496-1.					

Because the soluble MS recovery was within the QC limit range of 85-115% for aqueous samples, the hexavalent chromium result in the field blank was not subject to qualification (NJDEP, 2009; US EPA, 2010).

Duplicate Analysis (QC Limit: ≤ 20 %RPD)

The duplicate analysis was performed on one set of duplicate aqueous sample aliquots from non-client sample JC21504-2 associated with the PPG field blank. The difference between the duplicate aqueous sample aliquots for Cr+6 in the QC sample was 6.5%RPD, a value below the 20%RPD laboratory QC limit. Because the %RPD value for Cr+6 was below the QC limit for aqueous QC sample, the associated field blank result is acceptable and does not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with a blank spike recovery of 100% for the aqueous matrix, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the quantitation reports in the raw data. The following equation (NJDEP, 2009) was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/L)} = A \times E$$

Where: A = concentration from calibration curve (mg/L)
 E = Dilution (if necessary)

The non-detected hexavalent chromium concentration for Sample PPG174-FB04 (JC21496-1) was listed as < 0.010 mg/L on the reporting form and – 0.001 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/L)} = A \times E$$

$$\text{Cr}^{+6} \text{ (mg/L)} = -0.0012 \text{ mg/L} \times 1 = -0.0012 \text{ mg/L}$$

$$\text{Cr}^{+6} \text{ (mg/L)} = -0.001 \text{ mg/L} = < 0.010 \text{ mg/L}$$

After rounding to three significant figures, this verifies that the non-detected hexavalent chromium concentration of < 0.010 mg/L for Sample PPG174-FB04 was correctly reported.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

The field blank sample was observed to fall below the Eh-pH phase diagram line, thereby suggesting that the sample experiences conditions of a “reducing” environment. The Cr+6 sample result in a reducing sample matrix is not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing conditions. The field blank sample Cr+6 concentration is a non-detect result and is also not expected to increase.

Summary for Hexavalent Chromium Analysis – SDG JC21496

Since the QC requirements were met in the field blank analysis, no Cr+6 results were subject to qualification.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***"National Functional Guidelines for Inorganic Superfund Data Review"***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG **SDGs:** JC21496/JC21496A

1. Were the appropriate sample preservation requirements met?..... **Yes** No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? **Yes** No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes **No**
Indicate the identity of the samples and why.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes **No**
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? Yes **No**
If "Yes", include the number of samples and laboratory sample ID numbers.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... **Yes** No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... **Yes** No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No

9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**

If “yes”, please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds
of concern at the site met? **Yes** No

12. Were the QC Summary Forms reviewed?..... **Yes** No

13. Internal Standards acceptable..... **Yes** No

14. MS/MSD acceptable..... Yes **No**

15. Calibration summaries acceptable..... **Yes** No

16. Serial dilutions acceptable..... **Yes** No

17. Inorganic duplicates acceptable..... **Yes** No

18. LCS recovery acceptable..... **Yes** No

19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2 and 4 for QC details. Qualified sample results are presented in Table 3 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC21590/JC21590A
Sample Dates: June 6, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: July 12, 2016

This data validation (DV) report presents the data review and result qualifications for ten (10) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on June 6, 2016, for sample delivery group (SDG) JC21590, as well as JC21590A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC21590A and JC21590 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 10 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC21590-1A through JC21590-10A (inclusive)
- Hexavalent chromium ("NJ-") in Samples JC21590-1 through JC20639-10 (inclusive)
- Hexavalent chromium ("*NJ-") in reanalysis samples JC21590-1R through JC21590-10R (inclusive)

No other sample results in SDG JC21590A and JC21590 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except the antimony results in Samples JC21590-7A and JC21590-10A, while the hexavalent chromium (Cr+6) concentrations were below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Tables 4 and 9 of this DV report.

Sample Receipt

The ten (10) post-excavation soil samples collected June 6, 2016, were received intact and preserved appropriately the same day, June 6, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 5.0 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC21590A and JC21590

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-B26	JC21590-1A	6/6/2016	Soil	Metals
PPG174-MSD	JC21590-1AD	6/6/2016	Soil	Metals
PPG174-MS	JC21590-1AS	6/6/2016	Soil	Metals
PPG174-DUP	JC21590-2A	6/6/2016	Soil	Metals
PPG174-MAIN-B27	JC21590-3A	6/6/2016	Soil	Metals
PPG174-MAIN-B28	JC21590-4A	6/6/2016	Soil	Metals
PPG174-MAIN-B29	JC21590-5A	6/6/2016	Soil	Metals
PPG174-MAIN-B30	JC21590-6A	6/6/2016	Soil	Metals
PPG174-MAIN-B31	JC21590-7A	6/6/2016	Soil	Metals
PPG174-MAIN-B32	JC21590-8A	6/6/2016	Soil	Metals
PPG174-MAIN-B33	JC21590-9A	6/6/2016	Soil	Metals
PPG174-MAIN-B34	JC21590-10A	6/6/2016	Soil	Metals
PPG174-MAIN-B26	JC21590-1	6/6/2016	Soil	Cr+6
PPG174-MSD	JC21590-1D	6/6/2016	Soil	Cr+6
PPG174-MS	JC21590-1S	6/6/2016	Soil	Cr+6
PPG174-MAIN-B26	JC21590-1RT	6/6/2016	Soil	TOC, SS, Fe2+
PPG174-MAIN-DUP	JC21590-2	6/6/2016	Soil	Cr+6
PPG174-MAIN-B27	JC21590-3	6/6/2016	Soil	Cr+6
PPG174-MAIN-B28	JC21590-4	6/6/2016	Soil	Cr+6
PPG174-MAIN-B29	JC21590-5	6/6/2016	Soil	Cr+6
PPG174-MAIN-B30	JC21590-6	6/6/2016	Soil	Cr+6
PPG174-MAIN-B31	JC21590-7	6/6/2016	Soil	Cr+6
PPG174-MAIN-B32	JC21590-8	6/6/2016	Soil	Cr+6
PPG174-MAIN-B33	JC21590-9	6/6/2016	Soil	Cr+6
PPG174-MAIN-B34	JC21590-10	6/6/2016	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting the metals data is numbered JC21590A, while the data package for the hexavalent chromium analyses is numbered JC21590. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC21590 together with the supplemental total organic carbon (TOC), sulfide screen and ferrous iron. The samples data were validated for

the five target metals (antimony, chromium, nickel, thallium, and vanadium), as were the hexavalent chromium data, and supplemental TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC21590A and JC21590 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC21590A

The data validation of the metals analytical data in SDG JC21590A was reviewed for the following data quality items and a check mark (✓) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|-----------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Field duplicate sample analysis |
| √ Data qualifiers | √ Data package completeness |

The 10 post-excavation soil samples were analyzed for the 5 target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 10 samples of SDG JC21590A, the antimony results in two samples (JC21590-7A and JC21590-10A) exhibited a concentration above the IGWSSL of 6 milligrams per kilogram (mg/kg) for antimony. All remaining target metals results were less than the respective IGWSSL and SRS limits.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP94157 indicating possible matrix interference. The case narrative identified the serial dilution result being outside QC limits for antimony; however, the percent difference (%D) result was acceptable due to low initial sample concentration (< 50 times instrument detection limit [IDL]). All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC21590A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%Relative Percent Difference [RPD])

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for PPG QC batch sample, JC21590-1A, as identified in Table 2 below. These recoveries indicate possible matrix interference. Following the DV review, the sample

antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP94157 Ω	JC21590-1A	Antimony	47.8 %	48.6 %	NJ-	Low
QC Limits are 75-125% MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias. Ω – The samples associated with QC Batch MP94157 consist of JC21590-1A through -10A (inclusive).						

The antimony results in the ten affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 4.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots of JC21590-1A. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging from 0.8 – 2.6%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 98.5% - 105.0% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative identified the serial dilution result being outside QC limits for antimony, however, the percent difference (%D) result was acceptable due to the low initial sample concentration (< 50 times IDL). The remaining serial dilution results associated with the soil samples ranged from 0 – 6.6%D for the other four analytes, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Field Duplicate Sample Analysis (QC Limit: ≤ 50%RPD)

One set of field duplicate samples was collected as part of SDG JC21590A. Field duplicate sample collection and analysis can provide a determination of sampling representativeness and precision. Gross differences between field sample duplicates can be an indication of inconsistent sampling techniques or sample matrix complexities/non-homogeneity.

The advisory data validation guidelines for field duplicate soil sample analysis vary. There is no NJDEP DV guideline for qualifying field duplicate results (NJDEP, 2002). Recently, EPA has recommended qualifying field duplicate results that differ by more than 50%RPD or > 2 × Contract Required Quantitation Limit (CRQL) (USEPA, 2012), while the Field Sampling Plan for Hudson

County chromium sites lists a data quality objective (DQO) of 50%RPD for soil samples (AECOM, 2010).

The results for the analysis of the one pair of field duplicate samples are presented in Table 3, below. It is apparent that the results for the metals analytes that were detected in the field duplicate samples were very similar in the two field duplicate samples of PPG174-MAIN-B27 and, thus, are not subject to qualification as estimated concentrations, as the concentrations between field duplicate samples differed by less than 22%RPD, while the antimony and thallium results differed by less than two times the reporting limit value ($< 2 \times \text{CRQL}$).

Table 3. Comparison of Field Duplicate Soil Sample Results – SDG JB21590A

Analyte	PPG174-DUP (mg/kg)	PPG174-MAIN-B27 (mg/kg)	% RPD	DV Flag
Antimony	< 2.3 NJ-	2.3 NJ-	$< 2 \times \text{CRQL}$	-
Chromium	78.7	75.5	4.2	-
Nickel	27.1	23.1	15.9	-
Thallium	< 1.1	< 1.1	$< 2 \times \text{CRQL}$	-
Vanadium	38.9	31.3	21.6	-
Total Solids	84.5 %	84.6 %	----	-
mg/kg – milligrams per kilogram < – The analyte was not detected at the stated reporting limit NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. CRQL – The value representing the US EPA Contract Laboratory Program contract required quantitation limit, often represented by the reporting limit $< 2 \times \text{CRQL}$ – The difference between field duplicate results was less than two times the CRQL and meets QC requirements for sampling representativeness.				

Thus, the field duplicate sample results meet the QC limit for sampling representativeness and precision and are not subject to qualification

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The post-excavation soil sample analytical results for the samples of SDG JC21590A were found to be compliant with the analytical methods for the analysis of metals in the 10 post-excavation soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP94157 associated with the ten soil samples: JC21590-1A through JC21590-10A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 4.

Table 4. Summary of Qualified Sample Metals Results in SDG JC21590A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-B26	JC21590-1A	Antimony	2.7	NJ-
PPG174-DUP	JC21590-2A	Antimony	< 2.3	NJ-
PPG174-MAIN-B27	JC21590-3A	Antimony	2.3	NJ-
PPG174-MAIN-B28	JC21590-4A	Antimony	< 2.4	NJ-
PPG174-MAIN-B29	JC21590-5A	Antimony	< 2.4	NJ-
PPG174-MAIN-B30	JC21590-6A	Antimony	< 2.3	NJ-
PPG174-MAIN-B31	JC21590-7A	Antimony	7.7	NJ-
PPG174-MAIN-B32	JC21590-8A	Antimony	< 2.2	NJ-
PPG174-MAIN-B33	JC21590-9A	Antimony	< 2.1	NJ-
PPG174-MAIN-B34	JC21590-10A	Antimony	8.0	NJ-

Key:
mg/kg – milligrams per kilogram
< – The analyte was analyzed for, but was not detected above the stated reporting limit;
NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC21590

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the ten post-excavation soil samples. The soil samples were re-analyzed in a second QC batch in SDG JC21590.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements in the initial analysis.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- √ Data qualifiers
- Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Field duplicate sample analysis

Hexavalent chromium was detected in each of the ten post-excavation soil samples analyzed in SDG JC21590, with sample Cr+6 results less than the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg, with all results less than 4 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery in the initial analysis in QC Batch GP98128 was outside control limits, as well as in reanalysis QC Batch GP98156. The RPD value for the duplicate analysis in the analysis QC Batch GP98128 was above control limits, as was the re-analysis result. However, although the case narrative stated that the high RPD values

were due to possible sample non-homogeneity, the result of the initial analysis was deemed acceptable following the DV review because of the low sample and duplicate concentrations. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99998 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 103.8% to 104.3% for the QC batch associated with the analysis of 10 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP98128 associated with the 10 soil samples of this SDG, as presented below in Table 5. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP98128 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. The remaining spike recoveries were within the respective QC limits.

Table 5. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC21590

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98128 Ж	JC21590-1	Cr ⁺⁶ , soluble	61.6 %	NJ-	Low
GP98128 Ж	JC21590-1	Cr ⁺⁶ , insoluble	78.6 %	----	----
GP98128 Ж	JC21590-1	Cr ⁺⁶ , post-digestion spike	94.24 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ж – The samples associated with QC Batch GP98128 consist of JC21590-1 through -10 (inclusive).					

The Cr+6 results qualified for low spike recoveries are flagged with “NJ-” in accordance with DV guidelines which recommend qualification of Cr+6 results as estimated values when associated with MS recoveries between 50 – 75% (NJDEP, 2009). These qualified Cr+6 results of the initial analysis are tabulated below in Table 9, together with the qualified results from the re-analysis of this QC batch.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from Sample location JC21590-1 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-MAIN-B26) was listed as 200%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD DV advisory QC limit for technical review of soil sample data (US EPA, 2010; AECOM, 2010). However, since the concentrations were less than five times the reporting limit (or CRQL), the applicable QC limit is a difference less than twice the CRQL or less than twice the reporting limit. In this case, the difference of 0.83

mg/kg was less than twice the reporting limit of 0.48 and meets the QC requirement for analytical precision in soil samples. The difference between the values for redox potential (2.3%RPD) and pH (0.3%RPD) also displayed acceptable analytical precision results. Because the duplicate analysis result for Cr+6 was below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 92.5% and 97.3% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Field Duplicate Analysis (QC Limit ≤ 50%RPD)

The results for the analysis of one set of field duplicate samples are presented in Table 6, below. The difference for the low-level concentrations observed in the field duplicate samples from sampling location PPG174-MAIN-B27 differed by only 5.7%RPD, which is considerably below the QC limit of 50%RPD for soil samples (US EPA, 2012).

Table 6. Comparison of Field Duplicate Soil Sample Results – SDG JC21590

Analyte	PPG174-DUP (mg/kg)	PPG174-MAIN-B27 (mg/kg)	% RPD	DV Flag
Hex.Chromium	1.8 NJ-	1.7 NJ-	5.7 %	----
mg/kg – milligrams per kilogram NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.				

Thus, the field duplicate results for the field duplicate samples from PPG174-MAIN-B27 demonstrated very good sampling representativeness and precision, with field duplicate soil sample results differing by less than 6%RPD. No soil sample Cr+6 results were qualified for sampling representativeness issues.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (kg)
 - D = % Solids/100

E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-DUP (JC21590-2) was listed as 1.8 mg/kg on the reporting form and 0.0367 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0367 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00240 \text{ Kg} \times 84.5/100} = \frac{0.00367}{0.0020280} = 1.8097 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 1.8 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 1.8 mg/kg for Sample PPG174-DUP was correctly reported. This was the highest detected Cr+6 concentration of the ten detected results for the 10 soil samples of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Nine of the ten soil samples were observed to clearly fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentrations are also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total chromium concentrations are all less than 80 mg/kg, except for the 137 mg/kg total chromium result in JC21590-4 (1.5 mg/kg Cr+6), thereby making it highly unlikely that Cr+6 concentrations would increase to any significant degree, as observed in many other PPG data packages with total chromium concentrations below 600 mg/kg.

There was one sample, JC21590-10, which fell above the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of an “oxidizing” soil environment. Despite the possible oxidizing conditions in JC21590-10, the Cr+6 sample results in this soil sample are not expected to increase significantly in value because the corresponding total chromium concentration is only 15.7 mg/kg, thereby limiting any potential for oxidation of chromium to Cr+6 that could approach the SCC of 20 mg/kg.

Hence, based on the sample total chromium and Cr+6 concentrations, it is highly unlikely that any of the affected samples including those in the “reducing” zone would approach the SCC for Cr+6 of 20 mg/kg due to limitation created by the relatively low total chromium concentrations available for potential oxidation.

Cr+6 Re-analyses in SDG JC21590

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the re-analysis batch consisting of 10 soil samples are summarized in this section. The QC requirements were met during the reanalysis of samples JC21590-1R through -10R in QC Batch GP98156, including the calibrations ($r = 0.99992$, 91.4 – 94.0% CCV Recoveries), QC blanks, and blank spike analysis (86.8% and 89.3%). The soluble MS recovery was similar in magnitude to that in the initial analysis, but still below QC limits, while the insoluble MS recovery was considerably improved in the reanalysis. The post spike was slightly higher and still meeting QC limits, as detailed below.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the soluble MS recovery was still below QC limits, as observed below in Table 7. The soluble MS recovery in JC21590-1R was marginally lower compared to the initial soluble MS recovery to exhibit a potential low bias in the ability to recover Cr+6 from the sample matrix.

Table 7. Hexavalent Chromium Re-analysis MS Recovery Results – JC21590

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98156 X	JC21590-1R	Cr ⁺⁶ , soluble	59.6 %	NJ-	Low
GP98156 X	JC21590-1R	Cr ⁺⁶ , insoluble	92.4 %	----	----
GP98156 X	JC21590-1R	Cr ⁺⁶ , post-digestion spike	98.86 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium X – The samples associated with QC Batch GP98156 consist of JC21590-1R through -10R (inclusive).					

Since the soluble MS recovery in QC Batch GP98156 was below the QC limits (75-125%) and between 50% - 75%, the Cr+6 results for the samples in this QC batch are subject to qualification and are to be flagged as estimated values and flagged with “NJ-”, in consideration of DV guidelines (NJDEP, 2009). However, since the result of the duplicate analysis for the re-analyzed samples was outside control limits, the results are subsequently qualified with the DV qualifier combination “*NJ-.” These “qualified” Cr+6 results of the reanalysis are presented below in Table 9 together with the qualified (“NJ-”) results of the initial Cr+6 results.

Duplicate Sample Analysis (≤ 35%RPD soils)

The duplicate analysis was performed on one set of duplicate soil sample aliquots in the re-analysis. The difference between the duplicate soil sample aliquot concentrations for Cr+6 in sample JC21590-1R (PPG-MAIN-B26) was listed as 200%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD QC limit for soil samples (US EPA, 2010; AECOM, 2010). However since the sample concentrations were not above five times the reporting limit, the applicable QC limit becomes $\leq 2 \times CRQL$. The difference between JC21590-1R and its duplicate aliquot was 1.7 mg/kg, a value greater than twice the reporting limit of 0.48 mg/kg, as presented below in Table 8. A possible cause of the observed differences between the duplicate results may be attributable to sample non-homogeneity.

Table 8. Duplicate Analysis Results Outside QC Limits

QC Batch	QC Sample	Analyte	Original	Duplicate	Difference	DV Qualifier
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			Result (mg/kg)	(mg/kg)		
GP98156 ¥	JC21590-1R	Cr+6	0.0	1.7	> 2 × CRQL	*J
mg/kg – milligrams per kilogram QC Limit is 35%RPD * – Duplicate analysis not within control limits; indeterminate bias direction. J – The reported result is an estimated value. CRQL – The value representing the US EPA Contract Laboratory Program contract required quantitation limit, often represented by the reporting limit; > 2 × CRQL – The difference between field duplicate results was less than two times the CRQL and exceeds QC requirements. ¥ – The samples associated with QC Batch GP98156 consist of JC21590-1R through -10R (inclusive).						

Consequently, since the duplicate Cr+6 analysis exceeded the QC limit, the ten soil sample Cr+6 results are qualified as estimated values with an indeterminate bias and are to be flagged with the qualifier “*J” (NJDEP, 2009). Because the soluble MS recovery was below QC limits, and the samples are subject to qualification to be flagged with “NJ-”, the final qualification for the Cr+6 results of the re-analysis is a qualifier combination of “*NJ-”, as presented below in Table 9.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC21590-1RT (PPG174-MAIN-B26), a QC samples which was analyzed twice, initially with a detected Cr+6 concentration of 0.83 mg/kg before being non-detected in the re-analysis, results well below the SCC of 20 mg/kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. The total organic carbon (TOC) analysis was performed within the 14-day analytical holding time. In accordance with the method, these analyses were performed on the sample experiencing the low spike recoveries. A concentration of TOC (11,600 mg/kg) and the ferrous iron (Fe+2) with a result of 0.80 % were detected in the QC sample in JC21590-1RT, thereby indicating the likely presence of a “reducing” soil matrix in the soil sample, as suggested by the presence of this soil sample below the Eh-pH phase line, as are eight of the other nine soil samples of this SDG.

The only sample not falling well below the Eh-pH phase diagram line was JC21590-10, suggesting that the sample is present in an “oxidizing” soil matrix. However, this sample with a detected Cr+6 concentration of 1.4 mg/kg should not cause concern, because the total chromium concentration is only 15.7 mg/kg, thereby making it highly unlikely that this sample location could exhibit a Cr+6 result approaching the SCC of 20 mg/kg.

The “reducing” conditions in the soil matrix of JC21590-1 appear supported by the detected TOC concentration and the detected Fe⁺² data to support the results of the Eh-pH analyses.

Summary for Hexavalent Chromium Analysis – SDGs JC21590

The qualified soil sample results from the initial Cr+6 analysis in SDG JC21590 are presented below in Table 9 alongside those qualified results obtained from the reanalysis of the samples. Both sets of analytical Cr+6 results for samples JC12590-1 through -10 and their re-analysis are still both qualified as estimated values (NJ-) due to a potential low bias, as the soluble MS recovery of the second analysis exhibited a similar recovery in the re-analysis that was performed within the

30-day holding time. The insoluble MS recovery was considerably improved over the initial recovery result, with both still being within QC limits. Since the duplicate analysis result in the re-analysis was outside QC limits, the Cr+6 results of the re-analysis are qualified as estimated values and flagged with “*NJ-” suggesting a potential low bias in the results, as well as a potential variability in the analytical precision.

Table 9. Comparison of Qualified Cr+6 Results in JC21590 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC21590 Result (mg/kg)	DV Qualifier	JC21590-R Results (mg/kg)	DV Qualifier
PPG174-MAIN-B26	JC21590-1	Cr+6	0.83	NJ-	< 0.48	*NJ-
PPG174-DUP	JC21590-2	Cr+6	1.8	NJ-	0.48	*NJ-
PPG174-MAIN-B27	JC21590-3	Cr+6	1.7	NJ-	0.83	*NJ-
PPG174-MAIN-B28	JC21590-4	Cr+6	1.5	NJ-	4.4	*NJ-
PPG174-MAIN-B29	JC21590-5	Cr+6	0.83	NJ-	0.73	*NJ-
PPG174-MAIN-B30	JC21590-6	Cr+6	1.3	NJ-	1.7	*NJ-
PPG174-MAIN-B31	JC21590-7	Cr+6	0.82	NJ-	< 0.48	*NJ-
PPG174-MAIN-B32	JC21590-8	Cr+6	1.1	NJ-	0.8	*NJ-
PPG174-MAIN-B33	JC21590-9	Cr+6	0.63	NJ-	0.68	*NJ-
PPG174-MAIN-B34	JC21590-10	Cr+6	1.4	NJ-	< 0.48	*NJ-

mg/kg – milligrams per kilogram
 < – The analyte was analyzed for but was not detected above the stated reporting limit.
 * – Duplicate analysis not within control limits; indeterminate bias direction.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

Professional judgement was applied in qualifying the Cr+6 results in both analyses as estimated values (NJ-) due to a potential low bias, as suggested by the MS results tabulated above in Table 5 and Table 7 with soluble MS recoveries falling within the 50% - 75% range where DV guidelines recommend qualification of associated sample Cr+6 results. Thus, the qualification of these Cr+6 results as estimated is an approach consistent with DV guidelines (NJDEP, 2009), while the Cr+6 results of the re-analysis were additionally flagged with “*” to suggest that there may be a potential variability in the analytical precision in the re-analysis.

Although the samples were re-analyzed within the 30-day holding time, the Cr+6 concentrations differed slightly upon reanalysis. However, all Cr+6 sample results exhibited a Cr+6 values considerably below the SCC of 20 mg/kg, consistent with the redox state of the sample’s soil environment and the low total chromium concentrations.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
*	Duplicate analysis not within control limits; indeterminate bias direction.
J	The reported result is an estimated value.

N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

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US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

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ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC21590/JC21590A

- 1. Were the appropriate sample preservation requirements met?..... Yes No

- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

- 3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

- 5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The antimony results in Samples JC21590-7A and -10A exceeded the IGWSSL of 6 mg/kg.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

- 7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No

9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**

If "yes", please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds
of concern at the site met? Yes **No**

12. Were the QC Summary Forms reviewed?..... **Yes** No

13. Internal Standards acceptable..... **Yes** No

14. MS/MSD acceptable..... Yes **No**

15. Calibration summaries acceptable..... **Yes** No

16. Serial dilutions acceptable..... **Yes** No

17. Inorganic duplicates acceptable..... Yes **No**

18. LCS recovery acceptable..... **Yes** No

19. Other QC acceptable?..... **Yes** No

Provide a brief explanation, if applicable.

Refer to DV report tables 2, 3, 5, 6, 7, and 8 for QC details. Qualified sample results are presented in Tables 4 and 9 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC21931/JC21931A
Sample Dates: June 10, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: July 14, 2016

This data validation (DV) report presents the data review and result qualifications for twenty-two (22) post-excavation soil samples and one (1) field blank (FB) collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on June 10, 2016, for sample delivery group (SDG) JC21931A, as well as JC21931. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC21931A and JC21931 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 22 collected post-excavation soil samples and 1 field blank.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC21931-1, JC21931-7A through JC21931-20A (inclusive)

No other sample results in SDG JC21931A and JC21931 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except for the nickel results that were above the IGWSSL of 48 milligrams per kilogram (mg/kg) in samples JC21931-2A, -7A, -8A, -9A, -10A, -13A, , -18A, and -21A. The hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) of 20 mg/kg in SDG JC21931. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Table 4 of this DV report.

Sample Receipt

The 22 soil samples and 1 field blank collected June 10, 2016, were received intact and

appropriately preserved the same day, June 10, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 4.4 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC21931A and JC21931

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-B31R	JC21931-1	6/10/2016	Soil	Metals
PPG174-MAIN-CC05	JC21931-2A	6/10/2016	Soil	Metals
PPG174_FB06	JC21931-3A	6/10/2016	Aqueous	Metals
PPG174-MAIN-CC06	JC21931-4A	6/10/2016	Soil	Metals
PPG174-MAIN-CC07	JC21931-5A	6/10/2016	Soil	Metals
PPG174-MAIN-CC08	JC21931-6A	6/10/2016	Soil	Metals
PPG174 MAIN SW17	JC21931-7A	6/10/2016	Soil	Metals
PPG174 MAIN SW18	JC21931-8A	6/10/2016	Soil	Metals
PPG174 MAIN SW19	JC21931-9A	6/10/2016	Soil	Metals
PPG174 MAIN SW20	JC21931-10A	6/10/2016	Soil	Metals
PPG174 MAIN SW21	JC21931-11A	6/10/2016	Soil	Metals
PPG174 MAIN SW22	JC21931-12A	6/10/2016	Soil	Metals
PPG174 MAIN B35	JC21931-13A	6/10/2016	Soil	Metals
PPG174 MAIN B36	JC21931-14A	6/10/2016	Soil	Metals
PPG174 MAIN B37	JC21931-15A	6/10/2016	Soil	Metals
PPG174 MAIN B38	JC21931-16A	6/10/2016	Soil	Metals
PPG174 MAIN B39	JC21931-17A	6/10/2016	Soil	Metals
PPG174 MAIN SW23	JC21931-18A	6/10/2016	Soil	Metals
PPG174-MAIN-CC09	JC21931-19A	6/10/2016	Soil	Metals
PPG174 MAIN SW24	JC21931-20A	6/10/2016	Soil	Metals
PPG174 MAIN SW25	JC21931-21A	6/10/2016	Soil	Metals
PPG174-MAIN-CC10	JC21931-22A	6/10/2016	Soil	Metals
PPG174-MAIN-CC11	JC21931-23A	6/10/2016	Soil	Metals
PPG174-MAIN-CC05	JC21931-2	6/10/2016	Soil	Cr+6
PPG174_FB06	JC21931-3	6/10/2016	Aqueous	Cr+6
PPG174-MAIN-CC06	JC21931-4	6/10/2016	Soil	Cr+6
PPG174-MAIN-CC07	JC21931-5	6/10/2016	Soil	Cr+6
PPG174-MAIN-CC08	JC21931-6	6/10/2016	Soil	Cr+6
PPG174 MAIN SW17	JC21931-7	6/10/2016	Soil	Cr+6
PPG174 MAIN SW18	JC21931-8	6/10/2016	Soil	Cr+6
PPG174 MAIN SW19	JC21931-9	6/10/2016	Soil	Cr+6
PPG174 MAIN SW20	JC21931-10	6/10/2016	Soil	Cr+6
PPG174 MAIN SW21	JC21931-11	6/10/2016	Soil	Cr+6
PPG174 MAIN SW22	JC21931-12	6/10/2016	Soil	Cr+6
PPG174 MAIN B35	JC21931-13	6/10/2016	Soil	Cr+6
PPG174 MAIN B36	JC21931-14	6/10/2016	Soil	Cr+6
PPG174 MAIN B37	JC21931-15	6/10/2016	Soil	Cr+6
PPG174 MAIN B38	JC21931-16	6/10/2016	Soil	Cr+6
PPG174 MAIN B39	JC21931-17	6/10/2016	Soil	Cr+6
PPG174 MAIN SW23	JC21931-18	6/10/2016	Soil	Cr+6
PPG174-MAIN-CC09	JC21931-19	6/10/2016	Soil	Cr+6
PPG174 MAIN SW24	JC21931-20	6/10/2016	Soil	Cr+6
PPG174 MAIN SW25	JC21931-21	6/10/2016	Soil	Cr+6

PPG174-MAIN-CC10	JC21931-22	6/10/2016	Soil	Cr+6
PPG174-MAIN-CC11	JC21931-23	6/10/2016	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JC21931A, while the data package for the hexavalent chromium analyses is numbered JC21931.

Data Review

Data, as presented in the analytical data packages SDG JC21931A and JC21931 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002),
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP full deliverables package and is considered complete, as defined by the NJDEP “*Technical Regulations for Site Remediation*” (NJDEP, 2012). The data package was complete for the metals and hexavalent chromium analyses, such that the metals, Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC21931A

The data validation of the metals analytical data in SDG JC21931A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 22 post-excavation soil samples and 1 field blank were analyzed for the 5 target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids for the soil samples. Of the sample metals results detected in the 22 soil samples of SDG JC21931A, the nickel results in 8 samples were above the IGWSSL of 48 mg/kg in the following samples: JC21931-2A, -7A, -8A, -9A, -10A, -13A, -18A, and -21A.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP94240 indicating possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution result for antimony was outside control limits in QC Batch MP94240 associated with soil samples, while the result for chromium was outside control limits for QC Batch MP94236 associated with the field blank. However, the percent difference (%D) results were acceptable due to the low initial sample antimony and chromium concentrations (< 50 times instrument detection limit [IDL]). The detection limits for antimony, chromium, thallium and vanadium are elevated in JC21931-9A and -13A due to dilution required for high interfering element. The detection limits for chromium and thallium were elevated in JC21931-10A and -18A; and thallium in JC21931-20A, also due to dilution required for high interfering element. All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample and field blank analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

Exceptions included the 0% recovery of thallium in CRID1 at 12:16 and a 65% recovery of vanadium in CRID3 at 18:50 in analytical sequence MA39609 associated with the field blank and QC samples. However, the field blank results were not affected because the field blank was not associated with CRID1 since the field blank and QC samples were preceded by an acceptable CRID2 analysis. Additionally, the 50 micrograms per liter (µg/L) reporting limit for vanadium in the field blank is considerably above the affected range of 0 – 4 µg/L where results may be subject to

qualification. Hence, since the corresponding reporting limit is above the affected range of vanadium, the vanadium result in the field blank is also not subject to qualification.

The other exceptions consisted of the 135% recovery of thallium, in CRID1 at 11:26 and the 0% thallium recovery in CRID2 at 17:09 in analytical sequence MA39611 associated with most of the soil sample results. However, the soil sample results were not affected because thallium was not detected in any of the associated soil samples and a potential positive bias is not manifested in a non-detect result. Additionally, the non-detect thallium results in soil samples associated with the 0% CRID2 recovery are not subject to qualification because the thallium reporting limits of approximately 1 mg/kg are above the respective affected range of approximately 0 – 0.5 mg/kg for thallium.

Consequently, no soil sample or field blank results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks, the continuing calibration blanks (CCBs) or the field blank at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC21931A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for PPG QC batch sample JC21931-1, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits, including those of a non-client soil samples in QC Batch MP94235, and the non-client QC sample in QC Batch MP94236 associated with the field blank.

Table 2. Matrix Spike Recovery Results

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP94235 Ω	JC21765-8	Antimony	80.5 %	79.3 %	----	----
MP94236 ω	JC21912-1	Antimony	94.8 %	92.5 %	----	----
MP94240 §	JC21931-1	Antimony	54.9 %	56.4 %	NJ-	Low

QC Limits are 75-125%

MS – Matrix spike

MSD – Matrix spike duplicate.

NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.

Ω – The samples associated with QC Batch MP94235 consist of JC21931-2A, -4A, -5A, -6A, -21A, -22A,

and -23A;

ω – The sample associated with QC Batch MP94236 consists of JC21931-3A (field blank);

§ – The samples associated with QC Batch MP94240 consist of JC21931-1, JC21931-7A through -20A (inclusive).

The antimony results in the 15 affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 4.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on three pairs of spiked duplicate samples. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging from 0.0 – 3.3%RPD for soil samples and 0.0 – 2.4 %RPD for the batch QC sample associated with the field blank analysis with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 96.4% - 103.0% for the soil sample metals analysis, and 93.5 – 105.5% for the aqueous matrix.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative also stated that the RPD serial dilution result for antimony was outside control limits in QC Batch MP94240 associated with soil samples, while the result for chromium was outside control limits for QC Batch MP94236 associated with the field blank. However, the percent difference (%D) results were acceptable due to the low initial sample antimony and chromium concentrations (< 50 times IDL).

The remaining serial dilution results associated with the soil samples ranged from 0 – 6.8%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010), as well as 0 – 3.9%D for the aqueous fraction. No sample results required qualification for serial dilution issues.

Quantification Verification

Sample metals concentrations reported on the Form 1 sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported nickel results:

$$\text{Nickel (mg/kg)} = \frac{C \times V \times DF}{1000 \times W \times TS/100\%}$$

where:

- C = Raw instrument reading (µg/L)
- V = final volume (mL)
- DF = Dilution factor
- W = wet weight (gram [g])
- TS = Total solids (%)
- 1000 = conversion factor (milliliter per liter [mL/L])

The nickel concentration for Sample PPG174-MAIN-B35 (JC21931-13A) was listed as 17,000 mg/kg on the reporting form and 153.2 milligrams per liter (mg/L) on the quantitation report in the raw data for a 25-fold dilution. A calculation check provides the following result:

$$\text{Nickel (mg/kg)} = \frac{(153,200 \mu\text{g/L}) (100 \text{ mL}) (1)}{1000(\text{mL/L}) (1.02 \text{ g}) (88.6\%/100\%)} = \frac{15,320,000}{903.72} = 16,952.15 \mu\text{g/g}$$

$$= 17,000 \text{ mg/kg dry weight}$$

After rounding to three significant figures, this verifies that the nickel concentration of 17,000 mg/kg for Sample PPG174-MAIN-B35 was correctly reported. This nickel concentration was the highest detected metal result among the 22 soil samples of this SDG, a value clearly above the IGWSSL of 48 mg/kg for nickel.

Note that the software incorporates the dilution factor into the mg/L value in the raw data quantitation report, hence a dilution factor of only one (DF = 1) appears in the equation above, even though the dilution factor was 25× for nickel. The dilution factor appears as “Corr. Factor: 25.000” on the quantitation report header.

Reporting Limits

Sample JC21931-13A required a twenty-five-fold (25×) dilution for antimony, chromium, nickel, and thallium analysis, as well as 2× for vanadium, due to the presence of an interfering element, such that the antimony and thallium reporting limits for this sample were raised to values of < 55 and <28 mg/kg, values above the respective IGWSSLs of 6 and 3 mg/kg, as detailed below in Table 3. Because nickel was detected (17,000 mg/kg) above the IGWSSL of 48 mg/kg in this sample, the interpretation of the reporting limits for antimony and thallium are not affected by the elevated dilution, because the sample has to be addressed for the elevated nickel concentration.

Sample JC21931-9A required a five-fold (5×) dilution for antimony, chromium, thallium, and vanadium analysis due to the presence of an interfering element, such that the antimony and thallium reporting limits for this sample were raised to values of < 11 and < 5.4 mg/kg, values also above the respective IGWSSLs of 6 and 3 mg/kg, as detailed below in Table 3. Because nickel was detected at an elevated concentration (684 mg/kg) in this sample, interpretation of the antimony and thallium reporting limits for this sample are also not affected by the dilution.

Table 3. Sample Reporting Limits Affected by Sample Dilution

Sample ID	Lab ID	Analyte	Reporting Limit (mg/kg)	Dilution Factor	Adjusted Result (mg/kg)	Remediation Standard (mg/kg)
PPG174 MAIN SW19	JC21931-9A	Antimony	< 2.2	5	< 11	6
PPG174 MAIN SW19	JC21931-9A	Thallium	< 1.08	5	< 5.4	3
PPG174 MAIN B35	JC21931-13A	Antimony	< 2.2	25	< 55	6
PPG174 MAIN B35	JC21931-13A	Thallium	< 1.12	25	< 28	3

Units – milligrams per kilogram (mg/kg)

< - The analyte was analyzed for but was not detected above the stated reporting limit.

The interpretation of the elevated reporting limits for antimony and thallium in samples JC21931-9A and -13A was not compromised, because the nickel concentration was above the IGWSSL in the respective samples and these samples would need to be addressed in either additional review or some type of remedial action. Additionally, interpretation of the soil results was not compromised by these dilutions because thallium has not been detected in any of the soil samples during this phase of sampling at PPG.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC21931A were found to be compliant with the analytical methods for the analysis of metals in the 22 soil samples and 1 field blank using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP94240 associated with 15 of the 22 soil samples: JC21931-1, and JC21931-7A through JC21931-20A (inclusive). The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 4.

Table 4. Summary of Qualified Sample Metals Results in SDG JC21931A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-B31R	JC21931-1	Antimony	< 2.2	NJ-
PPG174 MAIN SW17	JC21931-7A	Antimony	< 2.1	NJ-
PPG174 MAIN SW18	JC21931-8A	Antimony	< 2.2	NJ-
PPG174 MAIN SW19	JC21931-9A	Antimony	< 11	NJ-
PPG174 MAIN SW20	JC21931-10A	Antimony	< 2.1	NJ-
PPG174 MAIN SW21	JC21931-11A	Antimony	< 2.2	NJ-
PPG174 MAIN SW22	JC21931-12A	Antimony	< 2.0	NJ-
PPG174 MAIN B35	JC21931-13A	Antimony	< 55	NJ-
PPG174 MAIN B36	JC21931-14A	Antimony	< 2.2	NJ-
PPG174 MAIN B37	JC21931-15A	Antimony	< 2.1	NJ-
PPG174 MAIN B38	JC21931-16A	Antimony	< 2.0	NJ-
PPG174 MAIN B39	JC21931-17A	Antimony	< 2.3	NJ-
PPG174 MAIN SW23	JC21931-18A	Antimony	< 2.2	NJ-
PPG174-MAIN-CC09	JC21931-19A	Antimony	< 2.0	NJ-
PPG174 MAIN SW24	JC21931-20A	Antimony	< 2.0	NJ-

Key:
 mg/kg – milligrams per kilogram
 < –The analyte was analyzed for but was not detected above the stated reporting limit.
 NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC21931

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in two QC batches for the 21 post-excavation soil samples and one QC batch for the field blank. There was no soil sample re-analysis because of the acceptability of the associated QC results.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Matrix spike recoveries
- √ Duplicate analysis

- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- √ Laboratory control samples
- √ Quantitation checks
- √ Data qualifiers

Hexavalent chromium was detected in 19 of the 21 soil samples analyzed in SDG JC21931, with all sample Cr+6 results less than 17 mg/kg, all values below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time, method blanks, as well as matrix spike recoveries. The case narrative did state that the RPD(s) for the duplicate Cr+6 analysis are outside control limits for Sample GP98252-D1 (JC21931-18) and that the high RPD may be due to possible sample non-homogeneity. No other QC requirements were exceeded.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibrations demonstrated acceptable correlation coefficients (“r”) with two values of 0.99977 for the soil samples analysis, as well as 0.99991 for the aqueous fraction, values greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 91.2% to 92.9% for QC Batch GP98252, 103.9% to 104.4% for QC Batch GP98251 associated with the analysis of 21 soil samples, and 104.7 to 105.0% for the aqueous fraction, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg), the continuing calibration blanks, or the field blank (< 0.010 mg/L). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble and insoluble matrix spike recoveries were all within the QC limits of 75-125% for the two QC batches associated with the 21 soil samples of this SDG and the QC batch associated with the field blank, as presented below in Table 5. Additionally, the post-digestion spike recoveries were within the QC limits of 85 – 115% demonstrating acceptable accuracy in the ability to recover Cr+6 from the sample matrices.

Table 5. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC21931

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GN47335 ω	JC21917-1	Cr ⁺⁶ , soluble	100.0 %	----	----
GP98251 ¥	JC21931-6	Cr ⁺⁶ , soluble	92.0 %	----	----
GP98251 ¥	JC21931-6	Cr ⁺⁶ , insoluble	109.8 %	----	----
GP98251 ¥	JC21931-6	Cr ⁺⁶ , post-digestion spike	102.26 %	----	----
GP98252 Ø	JC21931-18	Cr ⁺⁶ , soluble	78.8 %	----	----
GP98252 Ø	JC21931-18	Cr ⁺⁶ , insoluble	109.5 %	----	----
GP98252 Ø	JC21931-18	Cr ⁺⁶ , post-digestion spike	85.26 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery

MS – Matrix spike

Cr⁺⁶ – Hexavalent chromium

ω – The sample associated with QC Batch GN47335 consists of JC21931-3;

¥ – The samples associated with QC Batch GP98251 consist of JC21931-2, JC21931-4 through -13 (inclusive);

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
Ⓔ – The samples associated with QC Batch GP98252 consist of JC21931-14 through -23 (inclusive).					

Because all matrix spike recoveries were within the respective QC limits, no sample Cr+6 results were subject to qualification in the 21 soil samples and 1 field blank of this SDG.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on two sets of duplicate soil samples and one set of duplicate aqueous samples for a non-client QC batch sample associated with the field blank. The case narrative stated that the RPD(s) for the duplicate Cr+6 analysis are outside control limits for Sample GP98252-D1 (JC21931-18) and that the high RPD may be due to possible sample non-homogeneity. However, the difference between the duplicate soil sample aliquots for Cr+6 in soil sample JC21931-6 was 4.8 %RPD and the result for JC21931-18 was 28.6%RPD, a value above the 20%RPD laboratory QC limit, but below the 35%RPD QC limit for soil samples (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential were 1.6 and 4.1%RPD, and the pH results ranged from 0.6 to 2.0%RPD, displaying acceptable analytical precision results. Because the %RPD value for Cr+6 was below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries ranging 95.5% to 113.2% associated with the soil samples, and 106.7% for the aqueous matrix, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (Kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-MAIN-B39 (JC21931-17) was listed as 16.3 mg/kg on the reporting form and 0.347 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.347 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00245 \text{ Kg} \times 87.0/100} = \frac{0.03470}{0.0021315} = 16.2796 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 16.3 \text{ mg/kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 16.3 mg/kg for Sample PPG174-MAIN-B39 was correctly reported. This was the highest detected Cr+6 concentration of the 19 detected results for the 21 soil samples of this SDG analyzed for Cr+6, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Fourteen of the 21 soil samples were observed to fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a “reducing” soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions, while Cr+6 may increase under “oxidizing” conditions, provided there is a significant concentration of chromium available. Seven of the 21 soil samples were observed to fall above the Eh-pH phase line, thereby suggesting “oxidizing” conditions.

A review of the chromium to Cr+6 ratios for the samples of SDG JC21931 revealed that the Cr:Cr+6 ratios of samples falling within the “reducing” zone exhibited Cr:Cr+6 ratios ranging 10 to 500, while the samples falling above the Eh-pH phase line exhibited Cr:Cr+6 ratios ranging 4.3 to 22. Review of the chromium and Cr+6 results of the many analyzed soil samples for the PPG project suggests that generally the Cr:Cr+6 ratios tend to generally fall above a ratio of 20 to 1, which is not inconsistent with other studies in sites within New Jersey (Paustenbach, et al., 1991).

Summary for Hexavalent Chromium Analysis – SDG JC21931

Since the QC requirements were met in the soil samples and field blank analyses, no Cr+6 results were subject to qualification, thereby demonstrating acceptable accuracy and precision in the Cr+6 analysis.

The reported sample metals results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

Paustenbach, D.J., Rinehart, W.E., and P.J. Sheehan, 1991, "The Health Hazards Posed by Chromium-Contaminated Soils in Residential and Industrial Areas: Conclusions of an Expert Panel" in ***Regulatory Toxicology and Pharmacology***, 13, pp 195-222 (1991).

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***"National Functional Guidelines for Inorganic Superfund Data Review"***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC21931/JC21931A

1. Were the appropriate sample preservation requirements met?..... Yes No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Sample JC21931-9A was diluted 5× for antimony, chromium, thallium, and vanadium due to the presence of a high interfering element. Samples JC21931-10A and -18A were diluted 2× for chromium and thallium, while JC21931-20A was diluted 2× for thallium. Sample JC21931-13A was diluted 25× for antimony, chromium, nickel, and thallium, and 2× for vanadium. All were diluted due to the presence of a high interfering element.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

The reporting limits for antimony and thallium in Samples JC21931-9A and -13A exceeded the respective IGWSSL limits of 6 and 3 mg/kg.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The nickel results in the following samples exceeded the IGWSSL of 48 mg/kg: JC21931-2A, -7A, -8A, -9A, -10A, -13A, -18A, and -21A.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report Tables 2, 3, and 5 for QC details. Qualified sample results are presented in Table 4 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC22166/JC22166A
Sample Dates: June 14, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: July 21, 2016

This data validation (DV) report presents the data review and result qualifications for two (2) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on June 14, 2016, for sample delivery group (SDG) JC22166, as well as JC22166A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC22166A and JC22166 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 2 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC22166-1A and JC22166-2A

No other sample results in SDG JC22166A and JC22166 required qualification, based on the acceptability of the remaining associated quality control (QC) results and analytical performance. Details are provided in tables and text below. No hexavalent chromium results for the one soil sample of SDG JC22166 were qualified following the DV review, because all QC results were within method QC limits.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Table 3 of this DV report.

Sample Receipt

The two (2) soil samples collected June 14, 2016, were received intact and appropriately preserved the same day, June 14, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 5.2 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC22166A and JC22166

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-B34R	JC22166-1A	6/14/2016	Soil	Antimony
PPG174-MAIN-B40	JC22166-2A	6/14/2016	Soil	Metals
PPG174-MAIN-B40	JC22166-2	6/14/2016	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JC22166A, while the data package for the hexavalent chromium analysis is numbered JC22166.

Data Review

Data, as presented in the analytical data packages SDG JC22166A and JC22166 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16* (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced”

deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC22166A

The data validation of the metals analytical data in SDG JC22166A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

Soil sample JC22166-2A was analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), while JC22166-1A was analyzed for antimony, as indicated in Table 1, with both analyzed for percent total solids in the soil samples. Of the sample metals results detected in the samples of SDG JC22166A, no target analytes exceeded the respective IGWSSL or SRS.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP94278 indicating possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution result for thallium was outside control limits in this QC batch, however, the percent difference (%D) result was acceptable due to a low initial sample thallium concentration (< 50 times instrument detection limit [IDL]). All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample and field blank analyses and acceptable analyte quantitation (concentration determination) with the following exception.

The exception consisted of the 63.3% recovery of antimony in CRID1 at 10:53 in analytical sequence MA39633 associated with both soil samples. However, the soil sample results were not affected because the reporting limits for antimony are above the respective affected range where results may be subject to qualification. The affected range is approximately 0 – 0.67 mg/kg for antimony with corresponding reporting limits of approximately 2.1 mg/kg.

Thus, no sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC22166A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) recovery for antimony was below QC limits of 75 - 125% for the non-client QC sample in QC Batch MP94278 associated with the two soil samples which are summarized in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample results subject to qualification for a low spike recovery were flagged with “N” to indicate that the result is associated with QC recovery outside QC limits and were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP94278 Ω	JC21495-3R	Antimony	59.1 %	54.3 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias. Ω – The samples associated with QC Batch MP94278 consist of JC22166-1A and -2A.						

The antimony results in these two affected soil samples are flagged with “NJ-” due to a potential low bias. With the exception of the low-level detected thallium concentration, the metals concentrations in the non-client QC sample appear to be similar to those typically observed in PPG

samples and, therefore, qualification of the associated antimony results was judged appropriate in this case. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging from 1.8 – 11.4%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging 88.6% - 108.6% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative stated that the RPD serial dilution result for thallium was outside control limits in QC Batch MP94278, however, the percent difference (%D) result was acceptable due to a low initial sample concentration (< 50 times IDL). The remaining four serial dilution results associated with the soil samples ranged from 0.0 – 4.3%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limits.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC22166A were found to be compliant with the analytical methods for the analysis of metals in the 2 soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP94278 associated with the 2 soil samples: JC22166-1A and JC22166-2A. The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JC22166A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-B34R	JC22166-1A	Antimony	< 2.2	NJ-
PPG174-MAIN-B40	JC22166-2A	Antimony	< 2.1	NJ-
Key:				
mg/kg – milligrams per kilogram				
< –The analyte was analyzed for but was not detected above the stated reporting limit.				
NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and				

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
may experience a potential low bias.				

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC22166

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The one sample was analyzed in one QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | √ Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was detected in the single soil sample analyzed for Cr+6 in SDG JC22166, with a Cr+6 result of 0.59 mg/kg, a value clearly below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time, method blanks, as well as matrix spike recoveries. No QC requirements were exceeded.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99960 for the soil sample analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 95.7% to 95.9% for the QC batch associated with the analysis of the single soil sample, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The matrix spike and matrix spike duplicate (MS/MSD) recoveries for hexavalent chromium were all within QC limits of 75 - 125% for PPG sample JC22166-2, such that no soil sample results were qualified for matrix spike recoveries, thereby indicating acceptable analytical accuracy in the ability to recover Cr+6 in the associated sample matrices, as demonstrated in Table 4.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC22166

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98297 ¥	JC22166-2	Cr ⁺⁶ , soluble	79.0 %	----	----
GP98297 ¥	JC22166-2	Cr ⁺⁶ , insoluble	102.6 %	----	----
GP98297 ¥	JC22166-2	Cr ⁺⁶ , post-digestion spike	100.38 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium ¥ – The sample associated with QC Batch GP98297 consists of JC22166-2.					

Because of the acceptable MS recoveries, no Cr+6 results required qualification in the soil sample analysis.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from sample JC22166-2 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-MAIN-B40) was 3.4%RPD, a value below the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (7.2%RPD) and pH (0.9%RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 were below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 96.3% and 97.5% associated with the soil sample, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-MAIN-B40 (JC22166-2) was listed as 0.59 mg/kg on the reporting form and 0.0138 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0138 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00255 \text{ Kg} \times 92.8/100} = \frac{0.00138}{0.0023664} = 0.585 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 0.59 \text{ mg/kg}$$

After accounting for differences in rounding and rounding to two significant figures, this verifies that the hexavalent chromium concentration of 0.59 mg/kg for Sample PPG174-B40 was correctly reported. This was the only detected Cr+6 concentration of this SDG, a value clearly below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

The single soil sample was observed to fall below the Eh-pH phase diagram line, thereby suggesting that the sample experiences conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentration is also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total chromium concentration was detected as 17.5 mg/kg, thereby making it highly unlikely that the Cr+6 concentration would increase to any significant degree.

Summary for Hexavalent Chromium Analysis – SDG JC22166

Since the QC requirements were met in the soil sample analysis, no Cr+6 results were subject to qualification.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.

NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
------------	---

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG **SDGs:** JC22166/JC22166A

1. Were the appropriate sample preservation requirements met?..... **Yes** No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? **Yes** No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes **No**
Indicate the identity of the samples and why.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes **No**
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? Yes **No**
If "Yes", include the number of samples and laboratory sample ID numbers.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... **Yes** No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... **Yes** No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No

9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**

If “yes”, please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds
of concern at the site met? Yes **No**

12. Were the QC Summary Forms reviewed?..... **Yes** No

13. Internal Standards acceptable..... **Yes** No

14. MS/MSD acceptable..... Yes **No**

15. Calibration summaries acceptable..... **Yes** No

16. Serial dilutions acceptable..... **Yes** No

17. Inorganic duplicates acceptable..... **Yes** No

18. LCS recovery acceptable..... **Yes** No

19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report Tables 2 and 4 for QC details. Qualified sample results are presented in Table 3 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC22263
Sample Dates: June 15, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: July 28, 2016

This data validation (DV) report presents the data review and result qualifications for one (1) soil sample collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on June 15, 2016, for sample delivery group (SDG) JC22263. The sample was analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the sample of SDG JC22263 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the solitary collected soil sample.

Following the detailed DV review, the following sample results were qualified:

- Hexavalent chromium ("NJ-") in Sample JC22263-1
- Hexavalent chromium ("NJ-") in reanalysis sample JC22263-1R

No other sample results in SDG JC22263 required qualification, based on the acceptable remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below. The reported chromium and hexavalent chromium (Cr+6) concentrations were below the respective Soil Cleanup Criteria (SCC) limits. A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 4 of this DV report.

Sample Receipt

The one (1) soil sample collected June 15, 2016, was received intact and preserved appropriately the same day, June 15, at the Accutest laboratory in Dayton, NJ, with an acceptable sampling cooler temperature with a maximum corrected temperature of 4.1 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC22263

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-B06R2	JC22263-1U	6/15/2016	Soil	Chromium
PPG174-B06R2	JC22263-1	6/15/2016	Soil	Cr+6
PPG174-B06R2	JC22263-1RT	6/15/2016	Soil	TOC, SS, Fe2+
Chromium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential. TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.				

The data package presenting all of the data is numbered JC22263, which includes the chromium and the hexavalent chromium analysis. The data for the re-analysis of the sample for hexavalent chromium data are also found in JC22263 together with the supplemental total organic carbon (TOC), sulfide screen and ferrous iron. The sample data were validated for chromium, as were the hexavalent chromium data, and supplemental TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC22263 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16* (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data package in a NJDEP full deliverables package is considered complete, as defined by the NJDEP “*Technical Regulations for Site Remediation*” (NJDEP, 2012). The data package was complete for the chromium and hexavalent chromium analyses, and the chromium, Cr+6, and associated QC results were substantiated

during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC22263

The data validation of the metals analytical data in SDG JC22263 was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | √ Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The one soil sample was analyzed for total chromium, as well as percent total solids for the soil sample, and are covered by this data validation. Of the sample metals results detected in the sample of SDG JC22263, no result exhibited a concentration above the SRS..

Laboratory Case Narrative

The case narrative stated that all QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs) in most analytical sequences. Hence, no soil sample results warranted qualification for any associated QC blank contamination in SDG JC22263.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%Relative Percent Difference [RPD])

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for the chromium analysis were within the QC limits of 75 - 125% for non-client QC batch sample JC22593-3R in QC Batch MP94668, such that no sample metals results required qualification for matrix spike recovery results.

The metals results in the associated soil sample are not subject to qualification and the MS recovery results demonstrate acceptable accuracy.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. The %RPD value for chromium was below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with a value of 3.3%RPD for the solitary soil sample with no result requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with a blank spike recovery of 97.0% for the soil sample chromium analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The serial dilution result associated with the soil sample chromium analysis was 7.7%D, a value below the QC limit of 10%D criterion for data validation qualification (US EPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Sample metals concentrations reported on the Form 1 sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported chromium result:

$$\text{Chromium (mg/kg)} = \frac{C \times V \times DF}{1000 \times W \times TS/100\%}$$

- where:
- C = Raw instrument reading (micrograms per liter [μg/L])
 - V = final volume (milliliter [mL])
 - DF = Dilution factor
 - W = wet weight (gram [g])
 - TS = Total solids (%)
 - 1000 = conversion factor (milliliter per liter [mL/L])

The chromium concentration for Sample PPG174-B06R2 (JC22263-1U) was listed as 30.9 mg/kg on the reporting form and 0.2749 milligrams per liter (mg/L) on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Chromium (mg/kg)} = \frac{(274.9 \mu\text{g/L})(100 \text{ mL}) (1)}{1000(\text{mL/L}) (1.01 \text{ g}) (88.0\%/100\%)} = 30.9293 \mu\text{g/g}$$

$$= 30.9 \text{ mg/kg dry weight}$$

After rounding to 3 significant figures, this verifies that the chromium concentration of 30.9 mg/kg for Sample PPG174-B06R2 was correctly reported.

Thus, the analytical data presented met all QC requirements for these categories, with the exception of the issues discussed above.

Reporting Limits

The soil sample did not require dilution, such that the reporting limit for chromium was below the respective SCC value.

Summary of Qualified Metals Results

The soil sample analytical results for the one sample of SDG JC22263 were found to be compliant with the analytical methods for the analysis of metals in the single soil sample using SW-846 Method 6010C.

No soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC22263

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The single collected soil sample was analyzed in one QC batch and reanalyzed for Cr+6 in another QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Quantitation checks | √ Calibration verification |
| √ Data qualifiers | √ Data package completeness |

Hexavalent chromium was detected in the soil sample analyzed in SDG JC22263, and its re-analysis at the same concentration, with both Cr+6 results of 0.73 mg/kg, a value well below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery in QC Batch GP98324 was outside control limits, as was the soluble MS recovery in re-analysis QC Batch GP96362. The RPD value for the duplicate analysis in the initial analysis QC Batch GP98324 was above control

limits, however, the RPD value was acceptable due to low sample and duplicate concentrations. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99998 for the soil sample analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered at 92.2% for the QC batch associated with the initial analysis of single soil sample, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 mg/L). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP98324 associated with the single soil sample of this SDG, as presented below in Table 2. Thus, the hexavalent chromium results in the soil sample associated with QC Batch GP98324 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. All remaining MS recoveries were within QC limits.

Table 2. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC22263

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98324 ¥	JC22263-1	Cr ⁺⁶ , soluble	7.1 %	NJ-	Low
GP98324 ¥	JC22263-1	Cr ⁺⁶ , insoluble	77.2 %	----	----
GP98324 ¥	JC22263-1	Cr ⁺⁶ , post-digestion spike	95.27 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. ¥ – The sample associated with QC Batch GP98324 consists of JC22263-1.					

The Cr+6 result qualified for the low soluble matrix spike recovery is flagged with “NJ-”, as tabulated below in Table 4, together with the qualified result from the re-analysis of this QC batch.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from sample JC22263-1 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-B06R2) was 31.7%RPD, a value above the 20%RPD laboratory QC limit, but within the 35%RPD DV advisory QC limit for technical review of soil sample data (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (16.4%RPD) and pH (0.1%RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 were below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 81.8% and 81.6% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample PPG174-B06R2 (JC22263-1) was listed as 0.73 mg/kg on the reporting form and 0.0155 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0155 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00242 \text{ Kg} \times 88.0/100} = \frac{0.00155}{0.0021296} = 0.72784 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 0.73 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 0.73 mg/kg for Sample PPG174-B06R2 was correctly reported. This was the detected Cr+6 concentration of the single soil sample of this SDG, a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

The soil sample was observed to fall below the Eh-pH phase diagram line, thereby suggesting that the sample experienced conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentration is also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total chromium concentration in Sample JC22263-1 is only 30.9 mg/kg, thereby making it highly unlikely that the Cr+6 concentration would increase to any significant degree in a “reducing” soil matrix.

Summary for Hexavalent Chromium Analysis – SDG JC22263

Since the soluble MS spike recovery of 7.1% was below QC limits in the QC samples of QC Batch GP98324, the soil sample in this QC batch required re-analysis. The remaining QC results associated with the hexavalent chromium analysis were within QC limits. Therefore, the Cr+6 result for the sample of this QC batch in SDG JC22263 was qualified following the DV review and flagged with “NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix. Consequently, the soil sample of this QC batch was reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analyses in SDG JC22263.”

Cr+6 Re-analyses in SDG JC22263

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the batch consisting of 1 soil sample are summarized in this section. The QC requirements were met during the reanalysis of sample JC22263-1R in QC Batch GP98362, including the calibrations (r = 0.99997, 95.8 – 96.1% CCV Recoveries), QC blanks, duplicate analysis (4%RPD), and blank spike analysis (81.0% and 84.6%). The soluble and insoluble MS recoveries were considerably higher in the reanalysis, with the soluble MS recovery increasing from 7.1% to 59.1%, as detailed below.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the re-analysis of the affected sample. However, upon reanalysis, all but the post spike recovery in QC Sample JC22263-1R were all considerably higher compared to the initial analyses, particularly in the soluble spike, as observed below in Table 3. The insoluble MS recovery in JC22263-1R was still well within the 75-125% QC limits.

Table 3. Hexavalent Chromium Re-analysis MS Recovery Results – JC22263

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98362 Ж	JC22263-1R	Cr ⁺⁶ , soluble	59.1 %	NJ-	Low
GP98362 Ж	JC22263-1R	Cr ⁺⁶ , insoluble	95.2 %	----	----
GP98362 Ж	JC22263-1R	Cr ⁺⁶ , post-digestion spike	94.04 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium Ж – The sample associated with QC Batch GP98362 consists of JC22263-1R.					

Since the soluble MS recovery in QC Batch GP98362 was still below the QC limits (75-125%), the Cr+6 results for the samples in this QC batch are also subject to qualification as estimated values to be flagged with “NJ-” for a potential low bias in the ability to recover Cr+6 in this QC batch in accordance with DV guidelines (NJDEP, 2009). The Cr+6 result for the re-analysis of JC22263-1

(JC22263-1R) was qualified as an estimated result (0.73 NJ-), because the soluble MS recovery in the re-analysis was within the 50% - 75% QC range where DV guidelines recommend qualification of associated results (NJDEP, 2009). The qualified Cr+6 result of the re-analysis is presented below in Table 4 together with the result of the initial Cr+6 result.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC22263-1RT (PPG174-B06R2), a QC sample which was analyzed twice with detected concentrations of 0.73 mg/kg for both analyses, a value well below the SCC of 20 mg/kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. The total organic carbon (TOC) analysis was performed within the 14-day analytical holding time and, hence, the TOC result is not subject to qualification. In accordance with the method, these analyses were performed on the sample experiencing the low spike recoveries. A concentration of total organic carbon (55,300 mg/kg) and the ferrous iron (Fe+2) with a result of 0.51 % were detected in the QC sample in JC22263-1RT, thereby indicating the likely presence of a reducing soil matrix in the soil sample, as suggested by the presence of this soil sample below the Eh-pH phase line.

The “reducing” conditions in the soil matrix appear supported by the detected TOC concentration and the detected Fe⁺² data in support of the results of the Eh-pH analyses.

Summary for Hexavalent Chromium Analysis – SDGs JC22263

The qualified soil sample result from the initial Cr+6 analysis in SDG JC22263 is presented below in Table 4 alongside the qualified result obtained from the re-analysis of the sample. Both sets of analytical Cr+6 results for sample JC22263-1 are still both qualified as estimated values (NJ-) due to a potential low bias, as the soluble MS recovery of the second analysis exhibited a considerably higher recovery in the re-analysis that was performed within the 30-day holding time. The Cr+6 concentration determined during the re-analysis of Sample JC22263-1 was identical to that of the initial analysis, both still well below the SCC of 20 mg/kg.

Table 4. Comparison of Qualified Cr+6 Results in JC22263 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC22263 Result (mg/kg)	DV Qualifier	JC22263-R Results (mg/kg)	DV Qualifier
PPG174-B06R2	JC22263-1	Cr+6	0.73	NJ-	0.73	NJ-
mg/kg – milligrams per kilogram < –The analyte was analyzed for but was not detected above the stated reporting limit. NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.						

Professional judgement based on data usability considerations was applied in qualifying the Cr+6 results in both analyses as estimated values (NJ-) and not rejecting the Cr+6 result of the initial analysis for several reasons. Inorganic analyte data validation guidelines do not reject detected sample results (US EPA, 2014). NJDEP DV guidelines for Cr+6 analysis (NJDEP, 2009) recommend qualifying results associated with MS recoveries between 50-75% as estimated values to be flagged with “J” due to a potential low bias. Since the results of the initial and re-analysis were identical, despite the differences in MS recoveries, it was judged appropriate to qualify both results as estimated values (“NJ-”). The samples exhibited a corresponding total chromium

concentration of 31 mg/kg, making it highly improbable that the sample Cr+6 concentration would approach the SCC of 20 mg/kg for this samples experiencing “reducing” soil conditions. Additionally, the insoluble MS recoveries were within QC limits for both the initial and re-analyses and may be a better representation of the ability of the analysis to recover Cr+6 from the soil matrix than the soluble MS recovery result.

The Cr+6 results were qualified (“NJ-”), and not rejected based on: data usability considerations such as the results of the initial and re-analysis were identical detected Cr+6 concentrations; the insoluble MS recoveries were within the QC limits of 75-125%; the “reducing” soil environment of the sample; the abundance of TOC and Fe+2 supporting a “reducing” soil matrix; and especially the low total chromium content of only 31 mg/kg, thereby severely limiting the potential for oxidation of chromium to Cr+6 where the Cr+6 concentration might approach the SCC of 20 mg/kg.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

Kahn, Lloyd, 1988, ***Determination of Total Organic Carbon in Sediment (Lloyd Kahn Method)***, July 27, 1988: Edison, NJ, U.S. Environmental Protection Agency, Region II, 5 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, CLP, 2014, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9335.0-131, EPA-540-R-13-001, August 2014.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC22263

- 1. Were the appropriate sample preservation requirements met?..... **Yes** No

- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? **Yes** No
If "No", provide a brief explanation.

- 3. Were the samples diluted? Yes **No**
Indicate the identity of the samples and why.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable
standards?..... Yes **No**
If "Yes", list the affected samples.

- 5. Were any applicable standards exceeded for any samples? Yes **No**
If "Yes", include the number of samples and laboratory sample ID numbers.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for
the site?..... **Yes** No
If "No", provide a brief explanation of action taken.

- 7. Were qualifications noted in the non-conformance summary?..... **Yes** No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

- 8. Were qualified data used?..... **Yes** No

- 9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**
 If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
 Provide a brief explanation, if applicable.

Refer to DV report Tables 2 and 3 for QC details. Qualified sample results are presented in Table 4 of this DV report.



DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JC22737/JC22737A
Sample Dates: June 22, 2016
Analyses: Metals Analysis, EPA Method 6010C
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045C,D
Percent Solids, SM2540 G-97
Total Organic Carbon, Lloyd Kahn 1988 Mod.
Ferrous Iron, ASTM D3872-86
Sulfide Screen, SM4500S2-A-11
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: July 27, 2016

This data validation (DV) report presents the data review and result qualifications for two (2) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on June 22, 2016, for sample delivery group (SDG) JC22737, as well as JC22737A. The samples were analyzed for the analytes listed above employing the identified analytical methods by Accutest Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JC22737A and JC22737 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 2 collected post-excavation soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JC22737-1A and JC22737-2
- Hexavalent chromium ("NJ-") in Sample JC22737-1
- Hexavalent chromium ("NJ-") in reanalysis sample JC22737-1R

No other sample results in SDG JC22737A and JC22737 required qualification, based on the acceptable remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below.

The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, except the nickel result in Sample JC22737-2, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review and detail the affected samples whose results and reporting limits exceeded the respective standards or criteria.

The sample results that were subject to qualification following the DV review are presented in Tables 3 and 6 of this DV report.

Sample Receipt

The two (2) soil samples collected June 22, 2016, were received intact and appropriately preserved the same day, June 22, at the Accutest laboratory in Dayton, NJ, with acceptable sampling cooler temperatures with a maximum corrected temperature of 3.0 degrees Celsius. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JC22737A and JC22737

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
PPG174-MAIN-B41	JC22737-1A	6/22/2016	Soil	Metals
PPG174-MAIN-B35R	JC22737-2	6/22/2016	Soil	Sb, Ni, Tl
PPG174-MAIN-B41	JC22737-1	6/22/2016	Soil	Cr+6
PPG174-MAIN-B41	JC22737-1RT	6/22/2016	Soil	TOC, SS, Fe2+

Metals – Antimony (Sb), chromium, nickel (Ni), thallium (Tl) and vanadium analyzed by SW-846 Method 6010C at Accutest Laboratories in Dayton, NJ, as well as percent total solids.
Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.
TOC, SS, Fe2+ - The total organic carbon, sulfide screen and ferrous iron results were analyzed using methods detailed in the header of this DV report.

The data package presenting the metals data is numbered JC22737A, while the data package for the hexavalent chromium analyses is numbered JC22737. The data for the re-analysis of the samples for hexavalent chromium data are also found in JC22737 together with the supplemental total organic carbon (TOC), sulfide screen, and ferrous iron. The samples data were validated for the five target metals (antimony, chromium, nickel, thallium, and vanadium), as were the hexavalent chromium data, and supplemental TOC, sulfide screen and ferrous iron data.

Data Review

Data, as presented in the analytical data packages SDG JC22737A and JC22737, was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Data Review*”, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010 (US EPA, 2010).
- US EPA “*ICP-AES Data Validation, SOP No. HW-2a, Revision 15*” (USEPA, 2012).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JC22737A

The data validation of the metals analytical data in SDG JC22737A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The post-excavation soil sample JC22737-1A was analyzed for the five target EPA Method 6010C metals (antimony, total chromium, nickel, thallium, and vanadium), while JC22737-2 was analyzed for antimony, nickel and thallium, as well as percent total solids for the soil samples. Of the sample metals results detected in the 2 soil samples of SDG JC22737A, only nickel in JC22737-2 exceeded the IGWSSL of 48 milligrams per kilogram (mg/kg), while the remaining results were below the respective IGWSSL and SRS limits.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP94468 indicating possible matrix interference and/or sample non-homogeneity. The case narrative also stated that the relative percent difference (RPD) serial dilution result for nickel was outside control limits in QC Batch MP94468, however, the percent difference (%D) result was acceptable due to a low initial sample nickel concentration (< 50 times instrument detection limit [IDL]). All other QC requirements were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

The exceptions consisted of the 0% recovery of antimony in CRI1 at 11:29 and CRI3 at 16:07 in analytical sequence MA39705 associated with the two soil samples. However, the soil sample results were not affected because the reporting limits for antimony are above the affected range where results may be subject to qualification. The affected range is approximately 0 – 0.75 mg/kg for antimony where the corresponding reporting limits are 2.3 mg/kg. Thus, no soil sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < Continuing Calibration Verification Sample [CRDL] or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limits (RLs), such that no soil sample results warranted qualification for any associated QC blank contamination in SDG JC22737A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for a non-client QC batch sample JC22591-1, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results were further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2010; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP94468 Ω	JC22591-1	Antimony	69.6 %	68.4 %	NJ-	Low

QC Limits are 75-125%
MS – Matrix spike
MSD – Matrix spike duplicate.
NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.
Ω – The samples associated with QC Batch MP94468 consist of JC22737-1A and JC22737-2.

The antimony results in these two affected soil samples are flagged with “NJ-” due to a potential low bias in the ability to recover antimony from the sample matrix. The metals concentrations in the non-client QC sample appear to be similar to those typically observed in PPG samples and, therefore, qualification of the associated antimony results was judged appropriate in this case. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging from 0.7 – 5.4%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory Control Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 94.9% - 98.5% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit ≤ 10 %D)

The case narrative also stated that the RPD serial dilution result for nickel was outside control limits in QC Batch MP94468, however, the percent difference (%D) result was acceptable due to a low initial sample nickel concentration (< 50 times IDL). The remaining serial dilution results associated with the soil samples ranged from 0 – 4.3%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2014). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JC22737A were found to be compliant with the analytical methods for the analysis of metals in the 2 soil samples using SW-846 Method 6010C.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in QC Batch MP94468 associated with the 2 soil samples: JC22737-1A and JC22737-2. The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JC22737A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
PPG174-MAIN-B41	JC22737-1A	Antimony	< 2.3	NJ-
PPG174-MAIN-B35R	JC22737-2	Antimony	< 2.3	NJ-
Key: mg/kg – milligrams per kilogram < –The analyte was analyzed for but was not detected above the stated reporting limit.				

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
NJ- – The matrix spike recovery was below QC limits; associated sample result is estimated and may experience a potential low bias.				

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JC22737

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the single post-excavation soil sample. The soil sample was re-analyzed in a second QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was not detected in the single soil sample analyzed in SDG JC22737 or its re-analysis, hence meeting the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. The case narrative also stated that the soluble and insoluble matrix spike recoveries indicated possible matrix interference in QC Batch GP98493. However, the insoluble MS recovery (84.2%) was actually within QC limits. The soluble and insoluble matrix spike recoveries indicated possible matrix interference in re-analysis QC Batch GP98549, although the insoluble MS recovery of 74.9% may actually be considered acceptable when rounded to 2 significant figures (75-125%). All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification Sample [CCV] Recovery)

The initial calibration demonstrated an acceptable correlation coefficient (“r”) with a value of 0.99997 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered at 92.4% for the QC batch associated with the analysis of the one soil sample, both meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks (< 0.010 milligrams per liter [mg/L]). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP98493 associated with the single soil sample of this SDG, as presented below in Table 4. Thus, the hexavalent chromium result in the soil sample associated with QC Batch GP98493 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr+6 in the associated sample matrices. All remaining MS recoveries were within QC limits in the initial analysis.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JC22737

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98493 ¥	JC22737-1	Cr ⁺⁶ , soluble	5.4 %	NJ-	Low
GP98493 ¥	JC22737-1	Cr ⁺⁶ , insoluble	84.2 %	----	----
GP98493 ¥	JC22737-1	Cr ⁺⁶ , post-digestion spike	102 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
MS – Matrix spike
Cr⁺⁶ – Hexavalent chromium
NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
¥ – The sample associated with QC Batch GP98493 consists of JC22737-1.

The Cr+6 result qualified for low soluble MS recovery is flagged with “NJ-” (US EPA, 2014), as tabulated below in Table 6, together with the qualified result from the re-analysis of this QC batch.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil sample aliquots from sample JC22737-1 for the soil sample fraction. The difference between the duplicate soil sample aliquots for Cr+6 in this soil sample (PPG174-MAIN-B41) was 0.0%RPD, a value below the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (US EPA, 2010; AECOM, 2010), while the difference between the values for redox potential (0.9%RPD) and pH (0.9%RPD) also displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was below the QC limit for soil samples, the associated sample results are acceptable and do not warrant qualification. Hence, no Cr+6 sample results are subject to qualification for analytical precision issues.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 87.5% and 91.4% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr+6 results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)
 C = Wet weight of sample (Kg)
 D = % Solids/100
 E = Dilution (if necessary)

The non-detect hexavalent chromium concentration for Sample PPG174-MAIN-B41 (JC22737-1) was listed as < 0.47 mg/kg on the reporting form and 0.0060 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0060 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00246 \text{ Kg} \times 85.2/100} = \frac{0.00060}{0.0020959} = 0.2863 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 0.29 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the non-detect hexavalent chromium concentration of < 0.47 mg/kg for Sample PPG174-MAIN-B41 was correctly reported. This was the only sample analyzed for Cr+6 in this SDG, a value clearly below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

The solitary soil sample was observed to fall below the Eh-pH phase diagram line, thereby suggesting that the sample experiences conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The sample Cr+6 concentration is also not expected to increase to levels approaching the SCC of 20 mg/kg, because the total chromium concentration in JC22737-1 is only 17 mg/kg, thereby making it extremely unlikely that the Cr+6 concentration could increase to any significant degree.

Summary for Hexavalent Chromium Analysis – SDG JC22737

Since the soluble MS spike recovery was below QC limits in the QC sample of QC Batch GP98493, the soil sample in this QC batch required re-analysis. The remaining QC results

associated with the hexavalent chromium analysis were within QC limits. Therefore, the Cr+6 result for sample JC22737-1 was qualified following the DV review and flagged with “NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix. Consequently, the soil sample of this QC Batch GP98493 was reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analysis in JC22737”.

Cr+6 Re-analyses in SDG JC22737

Because the soluble MS recovery was below QC limits in the QC batch, the resultant data for the batch consisting of one soil sample is summarized in this section. The QC requirements were met during the re-analysis of sample JC22737-1R in QC Batch GP98549, including the calibrations (r = 0.99997, 97.4% CCV Recoveries), QC blanks, duplicate analysis (0 %RPD), and blank spike analysis (99.4% – 99.8%). The soluble MS recovery was considerably higher in the re-analysis, while the insoluble MS recovery was lower, as detailed below.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the re-analysis of the affected samples. However, upon re-analysis, the soluble MS recovery in QC Sample JC22737-1R was considerably better than in the initial analysis, but still under the QC limits, while the insoluble MS recovery was considerably lower falling to a value of 74.5%, just at the borderline of the QC limits of 75-125% after rounding to two significant figures, as observed below in Table 5. The post-digestion spike MS recovery fell significantly in the re-analysis but was still within the 85-115% QC limits.

Table 5. Hexavalent Chromium Re-analysis MS Recovery Results – JC22737

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP98549 X	JC22737-1R	Cr ⁺⁶ , soluble	44.0 %	NJ-	Low
GP98549 X	JC22737-1R	Cr ⁺⁶ , insoluble	74.9 %	----	----
GP98549 X	JC22737-1R	Cr ⁺⁶ , post-digestion spike	86.0 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
 MS – Matrix spike
 Cr⁺⁶ – Hexavalent chromium
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
 X – The sample associated with QC Batch GP98549 consists of JC22737-1R.

Since the soluble MS recovery in QC Batch GP98549 was still below the QC limits (75-125%), the Cr+6 result for the sample in this QC batch is also subject to qualification as an estimated value to be flagged with “NJ-” for a potential low bias in the ability to recover Cr+6 in this QC batch. The qualified Cr+6 result of the reanalysis is presented below in Table 6 together with the result of the initial Cr+6 analysis.

Supporting Analysis Results

The supporting analyses (ferrous iron, sulfide screen, and TOC) were analyzed on Sample JC22737-1RT (PPG174-MAIN-B41), a QC sample which was analyzed twice with non-detect concentrations of < 0.47 mg/kg for both analyses, values well below the SCC of 20 mg/kg. The ferrous iron and sulfide screen parameters were analyzed outside the respective holding times in order to provide more information about the possible impact of the sample matrix on the Cr+6 recoveries. The associated QC results were all within the respective QC limits. Professional

judgement was applied in not qualifying the affected sulfide screen and ferrous iron data. The total organic carbon (TOC) analysis was performed within the 14-day analytical holding time and, hence, the TOC result is not subject to qualification. In accordance with the method, these analyses were performed on the sample experiencing the low spike recoveries. A concentration of total organic carbon (219,000 mg/kg) and the ferrous iron (Fe+2) with a result of 0.57 % were detected in the QC sample in JC22737-1RT, thereby indicating the likely presence of a “reducing” soil matrix in the soil sample, as suggested by the presence of this soil sample below the Eh-pH phase line.

The “reducing” conditions in the soil matrix appear supported by the detected TOC concentration and the detected Fe⁺² data in support of the results of the Eh-pH analyses.

Summary for Hexavalent Chromium Analysis – SDGs JC22737

The qualified soil sample result from the initial Cr+6 analysis in SDG JC22737 is presented below in Table 6 alongside the qualified result obtained from the re-analysis of the single sample. Both sets of analytical Cr+6 results for Sample JC22737-1 and the re-analysis are still both qualified as estimated values (NJ-) due to a potential low bias, as the soluble MS recoveries were both below QC limits. The second analysis exhibited a considerably improved soluble MS recovery, while the insoluble MS recovery was lower, but at the lower QC limit of 75%. Both Cr+6 results, irrespective of the MS recovery values, were both non-detect concentrations.

Table 6. Comparison of Qualified Cr+6 Results in JC22737 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JC22737 Result (mg/kg)	DV Qualifier	JC22737-R Results (mg/kg)	DV Qualifier
PPG174-MAIN-B41	JC22737-1	Cr+6	< 0.47	NJ-	< 0.47	NJ-
<p>< –The analyte was analyzed for but was not detected above the stated reporting limit. NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.</p>						

Professional judgement was applied in qualifying the Cr+6 results in both analyses as estimated values (NJ-) due to a potential low bias, and not rejected, based on data usability considerations such as: the Cr+6 results of the initial and re-analysis were identical non-detect values; the insoluble MS recoveries were within and at the lower QC limit of 75-125%; the “reducing” soil environment of the sample; the abundance of TOC and Fe+2 supporting a “reducing” soil matrix; and especially the low total chromium content of only 17 mg/kg, thereby severely limiting the potential for oxidation of chromium to Cr+6 that might approach the SCC of 20 mg/kg.

Despite the changes in the soluble, insoluble and post-digestion MS recoveries between the initial and the re-analysis, performed within the 30-day holding time, Sample JC22737-1 exhibited identical non-detect Cr+6 results in both analyses.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, CLP, 2014, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9335.0-131, EPA-540-R-13-001, August 2014.

US EPA, 2012, ***ICP-AES Data Validation, SOP HW-2a, Revision 15***, December 2012.

US EPA, CLP, 2010, ***“National Functional Guidelines for Inorganic Superfund Data Review”***, OSWER Publication 9240.1-51, EPA540-R-10-011, January 2010.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JC22737/JC22737A

- 1. Were the appropriate sample preservation requirements met?..... **Yes** No

- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? **Yes** No
If "No", provide a brief explanation.

- 3. Were the samples diluted? Yes **No**
Indicate the identity of the samples and why.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes **No**
If "Yes", list the affected samples.

- 5. Were any applicable standards exceeded for any samples? **Yes** No
If "Yes", include the number of samples and laboratory sample ID numbers.

The nickel result in JC22737-2 exceeded the IGWSSL of 48 mg/kg.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... **Yes** No
If "No", provide a brief explanation of action taken.

- 7. Were qualifications noted in the non-conformance summary?..... **Yes** No
Provide a brief explanation.

Refer to DV report discussions of case narratives regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.
- Not applicable**
10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report Tables 2, 4, and 5 for QC details. Qualified sample results are presented in Tables 3 and 6 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDG JD13790/JD13790A
Sample Dates: September 28, 2020
Analyses: Metals Analysis, EPA Method 6010D
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: October 12, 2020

This data validation (DV) report presents the data review and result qualifications for six (6) soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on September 28, 2020, for sample delivery group (SDG) JD13790 and JD13790A. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD13790A and JD13790 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium, respectively, in the 6 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JD13790-1A through JD13790-6A (inclusive)

No other sample results in SDG JD13790A and JD13790 required qualification, based on the acceptable remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) in the respective SDGs, except for samples JD13790-5 and JD13790-6. A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 3 of this DV report.

Sample Receipt

The six (6) soil samples collected September 28, 2020 were received intact and appropriately

preserved the same day, September 28, 2020, at the SGS laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 2.8 degrees Celsius (°C). The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD13790A and JD13790

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
GGMB-1_2.0	JD13790-1A	9/28/2020	Soil	Metals
GGMB-1_4.0	JD13790-2A	9/28/2020	Soil	Metals
GGMB-1_6.0	JD13790-3A	9/28/2020	Soil	Metals
GGMB-1_8.0	JD137904A	9/28/2020	Soil	Metals
GGMB-1_10.0	JD13790-5A	9/28/2020	Soil	Metals
GGMB-1_12.0	JD13790-6A	9/28/2020	Soil	Metals
GGMB-1_2.0	JD13790-1	9/28/2020	Soil	Cr+6
GGMB-1_2.0 DUP	JD13790-1D	9/28/2020	Soil	Cr+6
GGMB-1_2.0 MS	JD13790-1S	9/28/2020	Soil	Cr+6
GGMB-1_4.0	JD13790-2	9/28/2020	Soil	Cr+6
GGMB-1_6.0	JD13790-3	9/28/2020	Soil	Cr+6
GGMB-1_8.0	JD13790-4	9/28/2020	Soil	Cr+6
GGMB-1_10.0	JD13790-5	9/28/2020	Soil	Cr+6
GGMB-1_12.0	JD13790-6	9/28/2020	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010D at SGS Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JD13790A, while the data package for the hexavalent chromium analyses is numbered JD13790.

Data Review

Data, as presented in the analytical data packages SDG JD13790A and JD13790 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on US EPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) *“National Functional Guidelines for Inorganic Superfund Methods Data Review”*, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA *“ICP-AES Data Validation, SOP No. HW-3a, Revision 1”* (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.

- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JD13790A

The data validation of the metals analytical data in SDG JD13790A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The six soil samples were analyzed for five target EPA Method 6010D metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids, which were covered by this data validation. Of the sample metals results detected in the 6 samples of SDG JD13790A, no sample metals results exhibited a concentration above the IGWSSL or SRS, whichever was more stringent.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP22993 indicating possible matrix interference and/or sample nonhomogeneity for the 6 soil samples analyzed in this SDG. The case narrative also identified the detection limit for thallium was elevated in samples JD13790-3A and JD13790-4A and for antimony in samples JD13790-5A and JD13790-6A due to a high

interfering element. The case narrative stated that the RPD serial dilution result for thallium was outside control limits in QC Batch MP22993, however, the percent difference (%D) results were acceptable due to a low initial sample concentrations (< 50 times instrument detection limit [IDL]). All other QC requirements were met for the target analytes of this SDG, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or < Reporting Limit [RL])

There were no target metals concentrations detected in the procedure blank or continuing calibration blanks (CCBs) at the stated reporting limits. Hence, no soil sample results warranted qualification for any associated QC blank contamination in SDG JD13790A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%Relative Percent Difference [RPD])

The matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were below the QC limits of 75 - 125% in the PPG QC sample JD13690-1 in QC Batch MP22993, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2017; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP22993 Ω	JD13690-1	Antimony	59.3 %	59.7 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias.						

Ω – The samples associated with QC Batch MP22993 consist of JD13790-1A through -6A (inclusive).

The antimony results in the six affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging from 0.7 – 2.1%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 89.5% – 97.0% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %Difference [%D])

The case narrative stated that the RPD serial dilution result for thallium was outside control limits in QC Batch MP22993, however, the percent difference (%D) results were acceptable due to a low initial sample concentration (< 50 times IDL). The serial dilution results for the remaining four analytes associated with the soil samples ranged from 0 – 5.3%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2017). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

The case narrative also identified the detection limit for thallium was elevated in samples JD13790-3A and JD13790-4A and for antimony in samples JD13790-5A and JD13790-6A due to a high interfering element. However, none of the reporting limits were above the respective IGWSSL or SRS values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JD13790A were found to be compliant with the analytical methods for the analysis of metals in the 6 soil samples using SW-846 Method 6010D.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in the QC batch associated with the 6 soil samples of this SDG, as detailed below in Table 3. The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias in the ability to recover antimony in the affected samples, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JD13790A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
GGMB-1_2.0	JD13790-1A	Antimony	< 2.3	NJ-

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
GGMB-1 4.0	JD13790-2A	Antimony	< 2.4	NJ-
GGMB-1 6.0	JD13790-3A	Antimony	< 2.3	NJ-
GGMB-1 8.0	JD13790-4A	Antimony	< 2.4	NJ-
GGMB-1 10.0	JD13790-5A	Antimony	< 23	NJ-
GGMB-1 12.0	JD13790-6A	Antimony	< 22	NJ-

Key:
 < –The analyte was analyzed for, but was not detected above the stated reporting limit.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JD13790

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the 6 soil samples.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- √ Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Data qualifiers

Hexavalent chromium was detected in five of the six soil samples analyzed in SDG JD13790, with samples JD13790-5 (297 mg/kg) and JD13790-6 (75.4 mg/kg) exceeding the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks and matrix spike recoveries. All QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification [CCV] Standard Recovery)

The initial calibration demonstrated an acceptable correlation coefficient ('r') with a value of 0.99995 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered with values of 91.8% for the QC batch associated with the 6 soil samples, thereby meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or <RL)

Hexavalent chromium was not detected in the method blank (< 0.40 mg/kg) or the continuing calibration blanks. Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The matrix spike (MS) recoveries for Cr+6 were all within the QC limits of 75 - 125% for PPG sample GGMB-1_2.0 (JD13790-1) in QC Batch GP30098, as depicted in Table 4, such that no sample Cr+6 results required qualification for matrix spike recovery results indicating acceptable analytical accuracy.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JD13790

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP30098 ¥	JD13790-1	Cr ⁺⁶ , soluble	84.8 %	----	----
GP30098 ¥	JD13790-1	Cr ⁺⁶ , insoluble	89.4 %	----	----
GP30098 ¥	JD13790-1	Cr ⁺⁶ , post-digestion spike	99.5 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
MS – Matrix spike
Cr⁺⁶ – Hexavalent chromium
¥ – The samples associated with QC Batch GP30098 consist of JD13790-1 through -6 (inclusive).

The Cr+6 results in the six associated soil samples are not subject to qualification and the MS recovery results demonstrate acceptable accuracy.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of duplicate samples. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with a value of 9.0 %RPD for soil samples with no results requiring qualification. The %RPD values for redox potential (13.7%RPD) and pH (2.7%RPD) displayed acceptable analytical precision results. The duplicate soil sample analyses demonstrated very good analytical precision.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 92.5% and 96.2% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
B = Final digested volume (L)

C = Wet weight of sample (kg)
D = % Solids/100
E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample GGMB-1_10.0 (JD13790-5) was listed as 297 mg/kg on the reporting form and 0.6657 mg/L on the quantitation report in the raw data for A 10-fold dilution. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.6657 \text{ mg/L} \times 0.1 \text{ L} \times 10}{0.00255 \text{ Kg} \times 87.8/100} = \frac{0.6657}{0.0022389} = 297.3335 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 297 \text{ mg/kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 297 mg/kg for Sample GGMB-1_10.0 was correctly reported. This was the highest detected Cr+6 concentration of the five detected results for the analysis of the 6 soil samples of this SDG, a value considerably above the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt (mV) solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Four of the 6 soil samples were observed to fall clearly below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. These four soil samples of this SDG contain total chromium concentrations of less than 400 mg/kg with a non-detect Cr+6 concentration in one sample and three detected concentrations less than 2 mg/kg.

The two remaining soil samples (JD13790-5 and JD13790-6) were above the Eh-pH phase line depicting oxidizing conditions and contained Cr+6 concentrations of 297 and 76.4 mg/kg with corresponding total chromium results of 6,460 and 3,200 mg/kg. Thus, the Cr+6 and total chromium results tend to reflect the conditions suggested by the Eh-pH plot.

Summary for Hexavalent Chromium Analysis – SDG JD13790

Since the MS recoveries were within QC limits, as were all other QC results associated with the hexavalent chromium analysis, including the duplicate sample analysis, no Cr+6 results were qualified following the DV review and are usable as reported.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, *Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey*, dated June 2010.

APHA, AWWA, and WEF, 1995, *Standard Methods for the Examination of Water and Wastewater, 19th Edition*, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, Trenton, New Jersey, April 2014.

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New Jersey Department of Environmental Protection, 2014c, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, Trenton, New Jersey, April 2014.

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New Jersey Department of Environmental Protection, 2012, *Technical Requirements for Site Remediation, N.J.A.C. 7:26E*, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10*, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on US EPA SW-846 Methods), SOP No. 5.A.16*, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, *Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4*, Trenton, New Jersey.

US EPA, CLP, 2017, *“National Functional Guidelines for Inorganic Superfund Methods Data Review”*, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017.

US EPA, 2016, *ICP-AES Data Validation, SOP HW-3a, Revision 1*, December 2016.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD13790/JD13790A

- 1. Were the appropriate sample preservation requirements met?..... Yes No

- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

- 3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Samples JD13790-3A and JD13790-4A were diluted 2× for thallium analysis, because of a high interfering element.

Samples JD13790-5A and JD13790-6A were diluted 10× for antimony, chromium, thallium, and vanadium analysis, because of a high interfering element.

Sample JD13790-5 was diluted 10× and Sample JD13790-6 was diluted 5× for Cr+6 analysis due to elevated Cr+6 concentrations.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

- 5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

Cr+6 concentrations exceeded the SCC of 20 mg/kg in samples JD13790-5 and -6.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
 Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... Yes No

9. Were rejections noted in the non-conformance summary?..... Yes No
 Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes No

If "yes", please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds
 of concern at the site met? Yes No

12. Were the QC Summary Forms reviewed?..... Yes No

13. Internal Standards acceptable..... Yes No

14. MS/MSD acceptable..... Yes No

15. Calibration summaries acceptable..... Yes No

16. Serial dilutions acceptable..... Yes No

17. Inorganic duplicates acceptable..... Yes No

18. LCS recovery acceptable..... Yes No

19. Other QC acceptable?..... Yes No
 Provide a brief explanation, if applicable.

Refer to DV report tables 2 and 4 for QC details. Qualified sample results are presented in Table 3 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JD13837/JD13837A
Sample Dates: September 29, 2020
Analyses: Metals Analysis, EPA Method 6010D
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga. Ph.D., REP5554
Report Date: October 26, 2020

This data validation (DV) report presents the data review and result qualifications for fourteen (14) soil samples and one (1) field blank (FB) collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey on September 29, 2020 for sample delivery group (SDG) JD13837, as well as JD13837A. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD13837A and JD13837 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 14 collected soil samples and one field blank.

Following the detailed DV review, the following sample results were qualified:

- Antimony (“NJ-”) in Samples JD13837-1A through JD13837-10A (inclusive), and JD13837-12A through JD13837-15A (inclusive);
- Chromium and vanadium (“EJ”) in Samples JD13837-1A through JD13837-10A (inclusive), and JD13837-12A through JD13837-15A (inclusive);
- Nickel, and thallium (“J”) in Samples JD13837-12A and JD13837-13A;
- Hexavalent chromium (“NR”) in Samples JD13837-1 through JD13837-10 (inclusive) and in Samples JD13837-12 through JD13837-15 (inclusive);
- Hexavalent chromium (“NR”) in reanalysis samples JD13837-1R through JD13837-10R (inclusive) and samples JD13837-12R through JD13837-15R (inclusive).

No other sample results in SDG JD13837A and JD13837 required qualification, based on the acceptable remaining associated QC results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Residential Soil Remediation Standard (SRS) and Impact to Groundwater Soil Screening Level (IGWSSL) limits, whichever was more stringent, except for vanadium in Sample JD13837-1A, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) of

20 mg/kg in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 5 and Table 9 of this DV report.

Sample Receipt

The fourteen (14) soil samples and one field blank collected September 29, 2020 were received intact and appropriately preserved September 29, 2020 at the SGS laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 3.2°C. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD13837A and JD13837

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
GGMB-1-14.0	JD13837-1A	9/29/2020	Soil	Metals
GGMB-1-16.0	JD13837-2A	9/29/2020	Soil	Metals
GGMB-1-18.0	JD13837-3A	9/29/2020	Soil	Metals
GGMB-1-20.0	JD13837-4A	9/29/2020	Soil	Metals
GGMB-1-22.0	JD13837-5A	9/29/2020	Soil	Metals
GGMB-1-24.0	JD13837-6A	9/29/2020	Soil	Metals
GGMB-2-1.0	JD13837-7A	9/29/2020	Soil	Metals
GGMB-2-3.0	JD13837-8A	9/29/2020	Soil	Metals
GGMB-2-5.0	JD13837-9A	9/29/2020	Soil	Metals
GGMB-2-9.0	JD13837-10A	9/29/2020	Soil	Metals
FB-01	JD13837-11A	9/29/2020	Aqueous	Metals
GGMB-2-11.0	JD13837-12A	9/29/2020	Soil	Metals
DUP01	JD13837-13A	9/29/2020	Soil	Metals
GGMB-2-13.0	JD13837-14A	9/29/2020	Soil	Metals
GGMB-2-23.0	JD13837-15A	9/29/2020	Soil	Metals
GGMB-1-14.0	JD13837-1	9/29/2020	Soil	Cr+6
GGMB-1-16.0	JD13837-2	9/29/2020	Soil	Cr+6
GGMB-1-18.0	JD13837-3	9/29/2020	Soil	Cr+6
GGMB-1-20.0	JD13837-4	9/29/2020	Soil	Cr+6
GGMB-1-22.0	JD13837-5	9/29/2020	Soil	Cr+6
GGMB-1-24.0	JD13837-6	9/29/2020	Soil	Cr+6
GGMB-2-1.0	JD13837-7	9/29/2020	Soil	Cr+6
GGMB-2-3.0	JD13837-8	9/29/2020	Soil	Cr+6
GGMB-2-5.0	JD13837-9	9/29/2020	Soil	Cr+6
GGMB-2-9.0	JD13837-10	9/29/2020	Soil	Cr+6
FB-01	JD13837-11	9/29/2020	Aqueous	Cr+6
GGMB-2-11.0	JD13837-12	9/29/2020	Soil	Cr+6
DUP01	JD13837-13	9/29/2020	Soil	Cr+6
GGMB-2-13.0	JD13837-14	9/29/2020	Soil	Cr+6
GGMB-2-23.0	JD13837-15	9/29/2020	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010D at SGS Laboratories in Dayton, NJ, as well as percent total solids.				
Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JD13837A, while the data package for the hexavalent chromium analyses is numbered JD13837.

Data Review

Data, as presented in the analytical data packages SDG JD13837A and JD13837 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Superfund Methods Data Review*”, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA “*ICP-AES Data Validation, SOP No. HW-3a, Revision 1*” (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JD13837A

The data validation of the metals analytical data in SDG JD13837A was reviewed for the following data quality items and a check mark (✓) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|---------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | Serial dilution analysis |
| √ ICP Interference Check Sample | Field duplicate sample analysis |
| √ Data package completeness | √ Data qualifiers |

The 14 soil samples and one field blank were analyzed for the five target EPA Method 6010D metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids, were covered by this data validation. Of the sample metals results detected in the 14 samples of SDG JD13837A, no results exhibited a concentration above the IGWSSL or SRS, whichever was more stringent, except for vanadium in Sample JD13837-1A.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC Batch MP23029 indicating possible matrix interference and/or sample nonhomogeneity in the soil samples analyzed in this SDG. The case narrative also stated that the RPD serial dilution result for antimony was outside control limits in QC Batch MP23029, however, the percent difference (%D) result was acceptable due to a low initial sample antimony concentration (< 50 times the instrument detection limit [IDL]). All other QC requirements for the analytes reviewed for data validation were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130%)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

The exception consisted of the 111.5% recovery of chromium in continuing calibration verification (CCV) standard CCV9 at 20:21 on 10/1/2020, a value above the QC limits of 90-110%. However, the soil sample results were not affected, as chromium was not reported for the samples associated with this CCV standard.

Hence, no sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks, the continuing calibration blanks (CCBs) or field blank at the stated reporting limit (RL) or contract required detection limit (CRDL). No soil sample results warranted qualification for any associated QC blank contamination in SDG JD13837A.

ICP Interference Check Samples (QC Limits: 80-120%)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the

specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis
(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were below the QC limits of 75 - 125% for the PPG QC batch sample JD13837-10A, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (USEPA, 2017; NJDEP, 2002). The remaining matrix spike results fell within QC limits, including those of QC Batch GP23033 for the field blank.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP23029 Ω	JD13837-10A	Antimony	58.2 %	58.2 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias. Ω – The samples associated with QC Batch MP23029 consist of JD13837-1A through -10A (inclusive) and JD13837-12A through -15A (inclusive).						

The antimony results in the 14 affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 5.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples from JD13837-10A. All %RPD values for the 5 target analytes were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 0.0 – 2.9%RPD for soil samples with no results requiring qualification and 0.0 – 4.0% for QC Batch MP23033 associated with the field blank. The duplicate analyses demonstrated very good analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging 80.8% - 93.1% for the soil sample metals analysis, and 93.5 – 101.5% for the aqueous fraction for the field blank analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative identified the serial dilution result being outside QC limits for antimony in QC Batch MP23029 and stated that the percent difference is acceptable due to low initial sample concentration (< 50 times IDL). However, it failed to identify that the serial dilution results for chromium and vanadium were outside QC limits and that the difference may indicate matrix interferences. Thus, the affected chromium and vanadium results in the fourteen samples

associated with this elevated %D exceedance are subject to qualification following the DV review, as discussed below. These QC results are detailed in Table 3 below.

Table 3. Serial Dilution Results Above QC Limits

QC Batch	QC Sample	Analyte	% Difference	DV Qualifier
MP23029 Ω	JD13837-10A	Chromium	11.6 %D	EJ
MP23029 Ω	JD13837-10A	Vanadium	10.6 %D	EJ
<p>Note: %D – Percent difference. EJ – The reported value is estimated because of the presence of interference; indeterminate bias direction. Ω – The samples associated with QC Batch MP23029 consist of JD13837-1A through -10A (inclusive) and JD13837-12A through -15A (inclusive).</p>				

The associated chromium and vanadium results in samples with laboratory sample ID numbers ranging JD13837-1A through -10A (inclusive) and JD13837-12A through -15A (inclusive), are qualified as estimated values and flagged with “EJ” to indicate that the result is an estimated value possibly experiencing variability in the reported value due to the presence of an interference in the sample matrix. The individual qualified results are presented in the summary table, Table 5, along with the results qualified for matrix spike recoveries outside QC limits.

Field Duplicate Sample Analysis (QC Limit ≤ 50%RPD)

One set of field duplicate samples were collected as part of SDG JD13837A. Field duplicate sample collection and analysis can provide a determination of sampling representativeness and precision. Gross differences between field sample duplicates can be an indication of inconsistent sampling techniques or sample matrix complexities/non-homogeneity.

The advisory data validation guidelines for field duplicate soil sample analysis vary. There is no NJDEP DV guideline for qualifying field duplicate results (NJDEP, 2002). Recently, EPA has recommended qualifying field duplicate results that differ by more than 50%RPD or > 2 × CRQL (USEPA, 2016), while the Field Sampling Plan for Hudson County chromium sites lists a data quality objective (DQO) of 50%RPD for soil samples (AECOM, 2010).

The results for the analysis of the one pair of field duplicate soil samples are presented in Table 4, below. It is apparent that the results for the soil metals analytes in the field duplicate samples of sample location GGMB-2-11.0 (JD13837-12A) were somewhat disparate with results differing by greater than 50%RPD or more than two times the reporting limit value (> 2 × CRQL) for all analyte pairs, thereby exceeding the data quality objective and demonstrating variable sampling representativeness and precision. These results are subject to qualification as estimated values to be flagged with “J”, as indicated below in Table 4.

Table 4. Comparison of Field Duplicate Soil Sample Results – SDG JD13837A

Analyte	GGMB-2-11.0 (mg/kg)	DUP01 (mg/kg)	% RPD	DV Flag
Antimony	< 12 NJ-	< 2.2 NJ-	> 2 × CRQL	(J)
Chromium	3,020 EJ	420 EJ	151 %	(J)
Nickel	490	288	51.9 %	J
Thallium	< 6.1	< 2.2	> 2 × CRQL	J
Vanadium	191 EJ	94.9 EJ	67.2 %	(J)
Total Solids	86.0 %	86.9 %	1.0 %	-

< – The analyte was not detected at the stated reporting limit;
 EJ – The reported value is estimated because of the presence of interference; indeterminate bias direction.
 (J) – Sample result is subject to DV qualification, but not additionally flagged with “J” to avoid redundancy;
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result may be biased low.
 CRQL – The value representing the US EPA CLP contract required quantitation limit, often represented by the reporting limit;
 > 2 × CRQL – The difference between field duplicate results was greater than two times the CRQL and exceeds QC requirements.

The field duplicate results for the field duplicate samples from GGMB-2-11.0 demonstrated variability in the sampling representativeness and precision, with field duplicate soil sample results differing by more than 50%RPD for the five target analytes. Thus, the soil sample results for nickel and thallium are qualified as estimated values and flagged with “J”. because the antimony results are already qualified as estimated values for a potential low bias (flagged with NJ-) and the chromium and vanadium results are qualified due to variability in the serial dilution analysis (flagged with “EJ”). The antimony, chromium, and vanadium results in these two field duplicate samples are not further qualified and flagged with “J” in order to avoid a redundancy of qualifiers that do not add value to the data evaluation process.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

Although various analytes in several samples were diluted for high interfering elements, no samples were diluted to the extent that the reporting limit exceeded the respective IGWSSL or SRS limit values.

Hence, all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results in JD13837A

The soil sample analytical results for the samples of SDG JD13837A were found to be compliant with the analytical methods for the analysis of metals in the 14 soil samples and one field blank using SW-846 Method 6010D.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in the QC batch associated with the 14 soil samples of this SDG, as detailed below in Table 5. The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias in the ability to recover antimony in the affected samples, as listed below in Table 5.

Table 5. Summary of Qualified Sample Metals Results in SDG JD13837A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
GGMB-1-14.0	JD13837-1A	Antimony	< 13	NJ-
GGMB-1-14.0	JD13837-1A	Chromium	3,710	EJ
GGMB-1-14.0	JD13837-1A	Vanadium	910	EJ
GGMB-1-16.0	JD13837-2A	Antimony	< 2.4	NJ-
GGMB-1-16.0	JD13837-2A	Chromium	753	EJ

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
GGMB-1-16.0	JD13837-2A	Vanadium	223	EJ
GGMB-1-18.0	JD13837-3A	Antimony	< 6.1	NJ-
GGMB-1-18.0	JD13837-3A	Chromium	575	EJ
GGMB-1-18.0	JD13837-3A	Vanadium	137	EJ
GGMB-1-20.0	JD13837-4A	Antimony	< 3.8	NJ-
GGMB-1-20.0	JD13837-4A	Chromium	373	EJ
GGMB-1-20.0	JD13837-4A	Vanadium	49.7	EJ
GGMB-1-22.0	JD13837-5A	Antimony	< 3.1	NJ-
GGMB-1-22.0	JD13837-5A	Chromium	67.9	EJ
GGMB-1-22.0	JD13837-5A	Vanadium	42.3	EJ
GGMB-1-24.0	JD13837-6A	Antimony	< 3.0	NJ-
GGMB-1-24.0	JD13837-6A	Chromium	36.4	EJ
GGMB-1-24.0	JD13837-6A	Vanadium	38.3	EJ
GGMB-2-1.0	JD13837-7A	Antimony	< 2.3	NJ-
GGMB-2-1.0	JD13837-7A	Chromium	28.4	EJ
GGMB-2-1.0	JD13837-7A	Vanadium	30.9	EJ
GGMB-3.0	JD13837-8A	Antimony	< 2.3	NJ-
GGMB-3.0	JD13837-8A	Chromium	31.7	EJ
GGMB-3.0	JD13837-8A	Vanadium	29.9	EJ
GGMB-5.0	JD13837-9A	Antimony	< 11	NJ-
GGMB-5.0	JD13837-9A	Chromium	125	EJ
GGMB-5.0	JD13837-9A	Vanadium	55.3	EJ
GGMB-2-9.0	JD13837-10A	Antimony	< 2.4	NJ-
GGMB-2-9.0	JD13837-10A	Chromium	207	EJ
GGMB-2-9.0	JD13837-10A	Vanadium	104	EJ
GGMB-2-11.0	JD13837-12A	Antimony	< 12	NJ-
GGMB-2-11.0	JD13837-12A	Chromium	3,020	EJ
GGMB-2-11.0	JD13837-12A	Nickel	490	J
GGMB-2-11.0	JD13837-12A	Thallium	< 6.1	J
GGMB-2-11.0	JD13837-12A	Vanadium	191	EJ
DUP01	JD13837-13A	Antimony	< 2.2	NJ-
DUP01	JD13837-13A	Chromium	420	EJ
DUP01	JD13837-13A	Nickel	288	J
DUP01	JD13837-13A	Thallium	< 2.2	J
DUP01	JD13837-13A	Vanadium	94.9	EJ
GGMB-2-13.0	JD13837-14A	Antimony	< 5.0	NJ-
GGMB-2-13.0	JD13837-14A	Chromium	1,280	EJ
GGMB-2-13.0	JD13837-14A	Vanadium	93.9	EJ
GGMB-2-23.0	JD13837-15A	Antimony	< 3.2	NJ-
GGMB-2-23.0	JD13837-15A	Chromium	63.4	EJ
GGMB-2-23.0	JD13837-15A	Vanadium	42.0	EJ

Key:

- < –The analyte was analyzed for, but was not detected above the stated reporting limit.
- J – The result is an estimated value;
- EJ – The reported value is estimated because of the presence of interference; indeterminate bias direction.
- NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JD13837

The analysis for hexavalent chromium (Cr⁺⁶) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the 14 soil samples and one QC batch for the field blank. The soil samples were re-analyzed in an additional QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|-----------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Field duplicate sample analysis |
| √ Data qualifiers | |

Hexavalent chromium was detected in two of the 14 soil samples analyzed in SDG JD13837, and three of the 14 reanalyzed samples, with all sample Cr+6 results less than 2 mg/kg, all values below the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble and insoluble matrix spike recoveries in QC Batch GP30148 were outside control limits, along with low post spike and pH-adjusted post spike recoveries, thereby suggesting that the recoveries indicate possible matrix interference. The soluble and insoluble matrix spike recoveries in reanalysis QC Batch GP30243 were also outside control limits, along with low post spike and pH-adjusted post spike recoveries. There was good agreement between the sample and 1:5 dilution in QC Batch GP30148 and GP30243. The RPD value for the duplicate analysis in reanalysis QC Batch GP30243 was outside control limits, but was acceptable due to low sample and duplicate concentrations. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% CCV Recovery)

The initial calibrations demonstrated acceptable correlation coefficients with a value of 0.99997 for the soil sample analysis and 0.99982 for the aqueous fraction, values greater than the calibration requirement for linearity of 0.995, as was the 0.99991 correlation coefficient in the reanalysis. The calibration check standard recoveries were 90.1% for the QC batch associated with the 14 soil samples and ranged from 99.5% to 101.2% for the aqueous fraction, all meeting the continuing calibration QC requirement of 90-110%. The calibration check standard recoveries were also 90.1% for the QC batch associated with the reanalysis of the 14 soil samples.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg), the continuing calibration blanks, or the field blank (< 0.010 mg/L). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble and insoluble matrix spike recoveries were below the QC limits of 75-125% for QC Batch GP30148 associated with 14 soil samples, with recoveries of 0% and 22.3%, respectively,

as presented below in Table 6. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP30148 are subject to rejection based on the results of the soluble MS and insoluble MS recoveries below 50%, as recommended in the DV guidelines for Cr+6 analysis (NJDEP, 2009) and the perceived inability to recover Cr+6 in the associated sample matrices. The post spike and pH-adjusted post spikes also recovered below QC limits such that the results are subject to qualification.

Table 6. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JD13837

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP30148 ¥	JD13837-10	Cr ⁺⁶ , soluble	0.0 %	NR	Low
GP30148 ¥	JD13837-10	Cr ⁺⁶ , insoluble	22.3 %	NR	Low
GP30148 ¥	JD13837-10	Cr ⁺⁶ , post-digestion spike	26 %	---	Low
GP30148 ¥	JD13837-10	Cr ⁺⁶ , pH-adjusted post spike	22 %	---	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium N – The matrix spike sample recovery in the associated QC sample is outside QC limits R – The result is rejected because the MS recovery in the associated QC sample is below 50%; ¥ – The samples associated with QC Batch GP30148 consist of JD13837-1 through -10 (inclusive), and JD13837-12 through -15 (inclusive).					

The Cr⁺⁶ results associated with MS recoveries below 50% are rejected and flagged with “NR”, as tabulated below in Table 9, together with the rejected results from the re-analysis of this QC batch.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JD13837-10. The difference between the duplicate soil sample aliquots for Cr+6 in soil this sample (GGMB-2-9.0) was listed as 0.0%RPD, a value below the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010). The RPD value for the aqueous QC Batch GN11853 associated with the field blank was also 0.0%. The %RPD values for redox potential (0.3%RPD) and pH (1.7%RPD) displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was within the QC limit for soil samples, the associated sample results are not subject to qualification and represent acceptable analytical precision.

Laboratory Control Sample Analysis (QC Limits: 80-120%)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 92.5% and 104.0% associated with the soil samples, and 98.0% for the aqueous fraction associated with the field blanks, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr⁺⁶ results were qualified for serial dilution analysis results. Serial dilution is not a requirement of the analytical method, although it appears that a serial dilution analysis was performed in the analytical sequence. A note on the MS Results Summary page indicated that there was good agreement between the sample and the 1:5 dilution in the reanalysis QC batch.

Field Duplicate Analysis (QC Limit ≤ 50%RPD)

The results for the analysis of one set of field duplicate samples are presented in Table 7, below. The difference for the low-level concentrations observed in the field duplicate samples from

sampling locations GGMB-2-11.0 differed by less than two times the reporting level, the QC limit when sample concentrations are less than five times the reporting limit, since the field duplicate sample results were both non-detect concentrations.

Table 7. Comparison of Field Duplicate Soil Sample Results.

Analyte	GGMB-2-11.0 (mg/kg)	DUP01 (mg/kg)	% RPD	DV Flag
Hex.Chromium	< 0.46 NR	< 0.46 NR	0%	-
<p>< – The analyte was not detected at the stated reporting limit. N – The matrix spike sample recovery in the associated QC sample is outside QC limits R – The result is rejected because the MS recovery in the associated QC sample is below 50%; NR – The matrix spike sample recovery in the associated QC sample is below the 50% QC limit recommending rejection of the result.</p>				

The field duplicate results from sampling location GGMB-2-11.0 are not subject to qualification because the difference between the results (0%RPD) met the data quality objective and QC limits for sampling, thereby indicating acceptable sampling representativeness and precision for the Cr+6 analysis.

Sample Result Verification

Sample Cr⁺⁶ concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr⁺⁶ results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (Kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample GGMB-2-13.0 (JD13837-14) was listed as 0.96 mg/kg on the reporting form and 0.0201 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0201 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00252 \text{ Kg} \times 83.3/100} = \frac{0.00201}{0.002092} = 0.9575 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 0.96 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 0.96 mg/kg for Sample GGMB-2-13.0 was correctly reported. This was the highest detected Cr+6 concentration of the two detected results for the initial analysis of the 14 soil samples of this SDG, a value below the SCC of 20 mg/kg. The Cr+6 results of the detected concentrations were less than 1.0 mg/kg in the initial analysis.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard mV solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Each of the 14 soil samples were observed to fall considerably below or near the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The total chromium concentrations of the samples falling below the Eh-pH phase line representing “reducing” soil conditions ranged from 28.4 mg/kg to 3,710 mg/kg, a rather wide range.

It is possible that the reducing soil environment contributed significantly to the poor MS recoveries observed in the initial soil analysis, conditions which do not favor oxidation of chromium to Cr+6. Despite the poor MS recoveries, it is possible that the reducing environment conditions exhibited by the Eh-pH phase diagram contribute significantly to the observed low or non-detect Cr+6 results and may actually reflect realistic representative results, as nine of the samples contain concentrations of total chromium that are less than 500 mg/kg (five less than 70 mg/kg) and are not expected to approach the SCC of 20 mg/kg for Cr+6 due to the reducing soil conditions.

Review of approximately 500 soil samples from PPG Site 63/65 has shown that samples containing 500 mg/kg might contain a corresponding Cr+6 concentration approaching the SCC criterion, though not exceeding it. Soil samples containing chromium and Cr+6 may be found to typically exhibit a ratio of approximately 20:1, similar to ratios observed at various Hudson County sites by Paustenbach, et. al (1991).

Summary for Initial Hexavalent Chromium Analysis – SDG JD13837

Since the soluble MS spike recovery of 0.0% was below QC limits in the QC samples of QC Batch GP30148, the soil samples in this QC batch required reanalysis. The 22.3% insoluble MS recovery was also below 50%, while the post spike and pH-adjusted post spike recoveries were below the respective QC limits, as depicted in Table 6. Therefore, the Cr⁺⁶ results for the 14 samples of this QC batch in SDG JD13837 were rejected following the DV review and flagged with “NR” due to a potential inability to recover hexavalent chromium from the soil sample matrix. Consequently, the soil samples of this QC batch were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analyses in SDG JD13837”.

Cr+6 Re-analyses in SDG JD13837

Because the soluble MS recovery was below QC limits in the initial QC batch triggering reanalysis, the resultant data for the reanalysis of the 14 soil samples (JD13837-1R through -10R, and JD13837-12R through -15R) are summarized in this section.

The QC requirements were met during the reanalysis of samples JD13837-1R through -10R and JD13837-12R through -15R in QC Batch GP30243, including the calibrations ($r = 0.99991$, 90.1% CCV Recoveries), QC blanks, duplicate analysis ($\leq 2 \times \text{CRQL}$), and blank spike analysis (91.5% – 103.1%). The – 1.0% soluble MS recovery in the reanalysis was similar to the 0% recovery in the

initial analysis, while the 48% insoluble MS recovery, post spike (49%) and pH-adjusted post spike (60%) recoveries were considerably improved, but still recovering below the respective QC limits, as detailed below in Table 8.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble MS and insoluble MS recoveries were still below 50% in the reanalysis, thereby confirming the decision to reject the Cr+6 sample results in both the initial and reanalysis. The low recoveries of the post-digestion spike and pH-adjusted post spike analysis support the observations of the soluble and insoluble MS recoveries, despite the improved recoveries for all but the soluble MS recovery.

The following matrix spike recoveries were observed during the reanalysis of the affected samples (Table 8).

Table 8. Hexavalent Chromium Re-analysis MS Recovery Results – JD13837

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP30243 X	JD13837-10R	Cr ⁺⁶ , soluble	Minus 1.0 %	NR	Low
GP30243 X	JD13837-10R	Cr ⁺⁶ , insoluble	48.0 %	NR	Low
GP30243 X	JD13837-10R	Cr ⁺⁶ , post-digestion spike	49 %	----	Low
GP30243 X	JD13837-10R	Cr ⁺⁶ , pH-adjusted post spike	60 %	---	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium X – The samples associated with QC Batch GP30243 consist of JD13837-1R through -10R (inclusive), and JD13837-12R through -15R (inclusive).					

Since the soluble and insoluble MS recoveries in QC Batch GP30243 are still below 50%, the Cr⁺⁶ results for the samples in this QC batch are also subject to rejection to be flagged with “NR” for a potential inability to recover Cr⁺⁶ in this QC batch. The qualified Cr+6 results of the reanalysis are presented below in Table 9 together with the results of the initial Cr+6 analysis.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JD13837-10R. The difference between the duplicate soil sample aliquots for Cr+6 in soil this sample (GGMB-2-9.0) was listed as 200.0%RPD, a value above the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010). However, the difference between the sample (0.44 mg/kg) and its duplicate (0.0 mg/kg) was a value less than two times the reporting limit of < 0.47mg/kg. The %RPD values for redox potential (0.3%RPD) and pH (1.9%RPD) displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was within the QC limit of “< 2 × 0.47” mg/kg for soil samples, the associated sample results are not subject to qualification and represent acceptable analytical precision.

Hence, the Cr+6 results in the associated samples were not qualified for the duplicate analysis result and analytical precision is considered acceptable in the re-analysis.

Summary for Hexavalent Chromium Analysis – SDGs JD13837

The qualified soil sample results from the initial Cr+6 analysis in SDG JD13837 are presented below in Table 9 alongside those qualified results obtained from the reanalysis of samples in this SDG. Both sets of analytical Cr+6 results for samples JD13837-1 through -10 (inclusive) and JD13837-12 through -15 (inclusive) and their reanalysis are still both rejected (“NR”) due to the very low spike recoveries and a potential low bias, as recommended by NJDEP DV guidelines (NJDEP, 2009). The Cr+6 concentrations determined during the re-analysis of samples in SDG JD13837, that were performed 7 days later within the 30-day holding time, differ only slightly from those of the initial analysis, being mostly non-detect results with the three detected results < 2 mg/kg, still well below the SCC of 20 mg/kg.

Table 9. Comparison of Qualified Cr+6 Results in JD13837 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JD13837 Result (mg/kg)	DV Qualifier	Reanalysis Results (mg/kg)	DV Qualifier
GGMB-1-14.0	JD13837-1	Cr+6	< 0.50	NR	< 0.50	NR
GGMB-1-16.0	JD13837-2	Cr+6	< 0.46	NR	0.50	NR
GGMB-1-18.0	JD13837-3	Cr+6	< 1.2	NR	< 1.1	NR
GGMB-1-20.0	JD13837-4	Cr+6	< 0.75	NR	< 0.76	NR
GGMB-1-22.0	JD13837-5	Cr+6	< 0.63	NR	< 0.64	NR
GGMB-1-24.0	JD13837-6	Cr+6	< 0.61	NR	< 0.60	NR
GGMB-2-1.0	JD13837-7	Cr+6	< 0.46	NR	< 0.46	NR
GGMB-3.0	JD13837-8	Cr+6	< 0.46	NR	1.1	NR
GGMB-5.0	JD13837-9	Cr+6	0.79	NR	1.9	NR
GGMB-2-9.0	JD13837-10	Cr+6	< 0.47	NR	< 0.47	NR
GGMB-2-11.0	JD13837-12	Cr+6	< 0.46	NR	< 0.45	NR
DUP01	JD13837-13	Cr+6	< 0.46	NR	< 0.46	NR
GGMB-2-13.0	JD13837-14	Cr+6	0.96	NR	< 0.48	NR
GGMB-2-23.0	JD13837-15	Cr+6	< 0.63	NR	< 0.65	NR

< –The analyte was analyzed for, but was not detected above the stated reporting limit.

N – The matrix spike sample recovery in the associated QC sample is outside QC limits

R – The result is rejected because the MS recovery in the associated QC sample is below 50%;

NR – The matrix spike sample recovery in the associated QC sample is below the 50% QC limit recommending rejection of the result.

Although the Cr+6 results were rejected in all 14 soil samples in the initial analysis, as well as the reanalysis in accordance with DV guidance (NJDEP, 2009), the guidance also suggests that the Eh-pH results can be referred to for data usability.

Despite the poor MS recoveries, it is possible that the reducing environment conditions exhibited by the Eh-pH phase diagram contribute significantly to the observed low or non-detect Cr+6 results and may actually reflect realistic representative results, as nine of the samples contain concentrations of total chromium that are less than 500 mg/kg with five samples less than 70 mg/kg Cr+6. These samples may not be expected to approach the SCC of 20 mg/kg for Cr+6 due to the reducing soil conditions.

Additionally, the results of the reanalysis were extremely similar to the low and non-detect results of the initial analysis, irrespective of the improved insoluble MS, post-digestion spike, and pH-adjusted post spike recoveries in the reanalysis, and seemingly irrespective of the total chromium concentration.

These results may be considered for use, with caution, in conjunction with other site information.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
E	This value is estimated due to the presence of interference, as indicated by the serial dilution analysis.
EJ	The reported value is estimated because of the presence of interference; indeterminate bias direction.
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
R	The result is rejected because the MS recovery in the associated QC sample is below 50%;

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

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US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD13837/JD13837A

1. Were the appropriate sample preservation requirements met?..... Yes No

2. Were appropriate sample holding times (for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Samples JD13837-1A, -12A, and -14A were diluted for antimony, chromium, thallium and vanadium analysis, while Sample JD13837-9A was diluted for antimony and thallium. Samples JD13837-4A, -8A, and -13A were diluted for thallium. These samples were diluted due to high interfering elements.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The concentration of vanadium in Sample JD13837-1A exceeded the SRS of 390 mg/kg.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... Yes No
9. Were rejections noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes No
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds of concern at the site met? Yes No
12. Were the QC Summary Forms reviewed?..... Yes No
13. Internal Standards acceptable..... Yes No
14. MS/MSD acceptable..... Yes No
15. Calibration summaries acceptable..... Yes No
16. Serial dilutions acceptable..... Yes No
17. Inorganic duplicates acceptable..... Yes No
18. LCS recovery acceptable..... Yes No
19. Other QC acceptable?..... Yes No
20. Provide a brief explanation, if applicable.

The field duplicate sample results for location GGMB-2-11.0 differed by more than the QC limits for all five metals (Table 4), with the results for nickel and thallium qualified strictly for the disparity between field duplicate samples and are flagged with “J” in Table 5.

Refer to DV report tables 2, 3, 4, 6, 7, and 8 for QC details. Qualified sample results are presented in Tables 5 and 9 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JD13913/JD13913A
Sample Dates: September 30, 2020
Analyses: Metals Analysis, EPA Method 6010D
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga. Ph.D., REP5554
Report Date: October 22, 2020

This data validation (DV) report presents the data review and result qualifications for eleven (11) soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey on September 30, 2020 for sample delivery group (SDG) JD13913, as well as JD13913A. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD13913A and JD13913 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 11 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JD13913-1A through JD13913-11A (inclusive);
- Hexavalent chromium ("NJ-") in Samples JD13913-1 through JD13913-11 (inclusive);
- Hexavalent chromium ("*NJ-") in re-analysis samples JD13913-1R through JD13913-11R (inclusive);

No other sample results in SDG JD13913A and JD13913 required qualification, based on the acceptable remaining associated QC results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Residential Soil Remediation Standard (SRS) and Impact to Groundwater Soil Screening Level (IGWSSL) limits, whichever was more stringent, except for vanadium in samples JD13913-10A and JD13913-11A, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC), except samples JD13913-9, -10, and -11, and re-analysis samples JD13913-9R, -10R, and -11R. A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 4 and Table 8 of this DV report.

Sample Receipt

The eleven (11) soil samples collected September 30, 2020 received intact and appropriately preserved September 30, 2020 at the SGS laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 2.9°C. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD13913A and JD13913

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
GGMB-5 1.0	JD13913-1A	9/30/2020	Soil	Metals
GGMB-5 3.0	JD13913-2A	9/30/2020	Soil	Metals
GGMB-5 5.0	JD13913-3A	9/30/2020	Soil	Metals
GGMB-5 7.0	JD13913-4A	9/30/2020	Soil	Metals
GGMB-5 9.0	JD13913-5A	9/30/2020	Soil	Metals
GGMB-5 11.0	JD13913-6A	9/30/2020	Soil	Metals
GGMB-5 13.0	JD13913-7A	9/30/2020	Soil	Metals
GGMB-5 15.0	JD13913-8A	9/30/2020	Soil	Metals
GGMB-5 17.0	JD13913-9A	9/30/2020	Soil	Metals
GGMB-5 19.0	JD13913-10A	9/30/2020	Soil	Metals
GGMB-5 21.0	JD13913-11A	9/30/2020	Soil	Metals
GGMB-5 1.0	JD13913-1	9/30/2020	Soil	Cr+6
GGMB-5 3.0	JD13913-2	9/30/2020	Soil	Cr+6
GGMB-5 5.0	JD13913-3	9/30/2020	Soil	Cr+6
GGMB-5 7.0	JD13913-4	9/30/2020	Soil	Cr+6
GGMB-5 9.0	JD13913-5	9/30/2020	Soil	Cr+6
GGMB-5 11.0	JD13913-6	9/30/2020	Soil	Cr+6
GGMB-5 13.0	JD13913-7	9/30/2020	Soil	Cr+6
GGMB-5 15.0	JD13913-8	9/30/2020	Soil	Cr+6
GGMB-5 17.0	JD13913-9	9/30/2020	Soil	Cr+6
GGMB-5 19.0	JD13913-10	9/30/2020	Soil	Cr+6
GGMB-5 21.0	JD13913-11	9/30/2020	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010D at SGS Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JD13913A, while the data package for the hexavalent chromium analyses is numbered JD13913.

Data Review

Data, as presented in the analytical data packages SDG JD19313A and JD19313 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16* (NJDEP, 2002).

- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Superfund Methods Data Review*”, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA “*ICP-AES Data Validation, SOP No. HW-3a, Revision 1*” (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JD19313A

The data validation of the metals analytical data in SDG JD19313A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The 11 soil samples were analyzed for the five target EPA Method 6010D metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids, were covered by this

data validation. Of the sample metals results detected in the 11 samples of SDG JD13913A, only the vanadium results in samples JD19313-10A and JD19313-11A exhibited a concentration above the IGWSSL or SRS, whichever was more stringent.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC Batch MP23031 indicating possible matrix interference and/or sample nonhomogeneity in the soil samples analyzed in this SDG. Samples JD13913-5A, -6A, -7A, and -8A were diluted for thallium analysis, while samples JD13913-9A and -10A were diluted for the analysis of four analytes (antimony, chromium, thallium, and vanadium) due to high interfering elements. Sample JD13913-11A was diluted for each of the five analytes (antimony, chromium, nickel, thallium, and vanadium), all due to high interfering elements. All other QC requirements for the analytes reviewed for data validation were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130%)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

Hence, no sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks and the continuing calibration blanks (CCBs) at the stated reporting limit (RL) or contract required detection limit (CRDL).

No soil sample results warranted qualification for any associated QC blank contamination in SDG JD19313A.

ICP Interference Check Samples (QC Limits: 80-120%)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis (QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for the non-client QC batch sample JD13899-7, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a

QC recovery outside QC limits and the antimony results further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (USEPA, 2017; NJDEP, 2002). The remaining matrix spike results fell within QC limits in Batch MP23031.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP23031 Ω	JD13899-7	Antimony	54.1 %	54.1 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias. Ω – The samples associated with QC Batch MP23031 consist of JD13913-1A through -11A (inclusive).						

The antimony results in the 11 affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in summary table, Table 4.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples from non-client sample JD13899-7. All %RPD values for the 5 target analytes were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 0.0 – 0.9%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated excellent analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging 91.3% - 94.9% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The serial dilution results associated with the soil samples ranged 0 – 3.1%D, values below the QC limit of 10%D criterion for data validation qualification (USEPA, 2010). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

The case narrative identified that the detection limits for various analytes were elevated in several samples due to a high interfering element.

There were two samples that required dilution that resulted in elevated reporting limits above the SRS of 31 mg/kg for antimony. Sample JD13913-10A was diluted by a factor of 20 and by a factor

of 50 in Sample JD13913-11A, thereby raising the antimony reporting limit to higher than the SRS of 31 mg/kg, as detailed below in Table 3, to compensate for the interfering element presence.

Table 3. Sample Reporting Limits Affected by Sample Dilution

Sample ID	Lab ID	Analyte	Reporting Limit (mg/kg)	Dilution Factor	Adjusted Result	Remediation Standard
GGMB-5 19.0	JD13913-10A	Antimony	< 2.5	20	< 50	31
GGMB-5 21.0	JD13913-11A	Antimony	< 2.4	50	< 120	31

Units – mg/kg
 < - The analyte was analyzed for, but was not detected above the stated reporting limit.

The interpretation of the reporting limits for antimony in samples JD13913-10A and JD13913-11A was not compromised because the vanadium concentration was above the SRS of 390 mg/kg and the samples would need to be addressed in either additional review or some type of remedial action.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JD13913A were found to be compliant with the analytical methods for the analysis of metals in the 11 soil samples using SW-846 Method 6010D.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in the QC batch associated with the 11 soil samples of this SDG, as detailed below in Table 4. The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias in the ability to recover antimony in the affected samples, as summarized below in Table 4.

Table 4. Summary of Qualified Sample Metals Results in SDG JD13913A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
GGMB-5 1.0	JD13913-1A	Antimony	< 2.3	NJ-
GGMB-5 3.0	JD13913-2A	Antimony	< 2.5	NJ-
GGMB-5 5.0	JD13913-3A	Antimony	< 2.2	NJ-
GGMB-5 7.0	JD13913-4A	Antimony	< 2.4	NJ-
GGMB-5 9.0	JD13913-5A	Antimony	< 2.2	NJ-
GGMB-5 11.0	JD13913-6A	Antimony	< 2.2	NJ-
GGMB-5 13.0	JD13913-7A	Antimony	< 2.5	NJ-
GGMB-5 15.0	JD13913-8A	Antimony	< 2.5	NJ-
GGMB-5 17.0	JD13913-9A	Antimony	< 12	NJ-
GGMB-5 19.0	JD13913-10A	Antimony	< 50	NJ-
GGMB-5 21.0	JD13913-11A	Antimony	< 120	NJ-

Key:
 < –The analyte was analyzed for, but was not detected above the stated reporting limit.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JD13913

The analysis for hexavalent chromium (Cr⁺⁶) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the 11 soil samples. The soil samples were re-analyzed in an additional QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

√ Holding times	Matrix spike recoveries
√ Blank Analysis	Duplicate analysis
√ Calibration standards	√ Laboratory control samples
√ Calibration verification	√ Quantitation checks
√ Data package completeness	√ Data qualifiers

Hexavalent chromium was detected in each of the 11 soil samples analyzed initially in SDG JD13913, and four of the 11 reanalyzed samples. The Cr+6 results in samples JD13913-9, -10, and -11 each exceeded the soil cleanup criterion (SCC) of 20 mg/kg, as did the results of their reanalysis. The Cr+6 results of the remaining eight samples were all less than 4 mg/kg when detected.

Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery in QC Batch GP30170 was outside control limits, while the soluble MS recovery in reanalysis QC Batch GP30317 was also outside control limits. The RPD value for the duplicate analysis in QC Batch GP30170 was outside control limits, as was the RPD value for the reanalysis batch, but the former was considered acceptable following DV review since the RPD value was less than 35%. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% CCV Recovery)

The initial calibration demonstrated an acceptable correlation coefficient with a value of 0.99992 for the soil sample analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 99.0% to 99.2% for the QC batch associated with the 11 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks. Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP30170 associated with 11 soil samples, as presented below in Table 5. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP30170 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr⁺⁶ in the associated sample matrices. The insoluble MS recovery was within QC limits and the post spike also recovered within QC limits.

Table 5. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JD13913

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP30170 ¥	JD13913-1	Cr ⁺⁶ , soluble	59.4 %	NJ-	Low
GP30170 ¥	JD13913-1	Cr ⁺⁶ , insoluble	85.8 %	----	----
GP30170 ¥	JD13913-1	Cr ⁺⁶ , post-digestion spike	98.9 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. ¥ – The samples associated with QC Batch GP30170 consist of JD13913-1 through -11 (inclusive).					

The Cr⁺⁶ results qualified for the low spike recovery are flagged with “NJ-”, as tabulated below in Table 8. Qualification of these Cr+6 results is discussed in the Summary at the end of the Cr+6 review.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JD13913-1. The difference between the duplicate soil sample aliquots for Cr+6 in soil this sample (GGMB-5_1.0) was listed as 34.5%RPD, a value above the 20%RPD laboratory QC limit, but below the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010). Additionally, the difference between the sample (1.7 mg/kg) and its duplicate (1.2 mg/kg) was a value less than two times the reporting limit of < 0.49 mg/kg, the QC limit when either concentration is less than five times the reporting limit. The %RPD values for redox potential (0.6%RPD) and pH (0.9%RPD) displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was within the QC limit of “< 2 × 0.49” mg/kg for soil samples, the associated sample results in the initial analysis are not subject to qualification and represent acceptable analytical precision.

Laboratory Control Sample Analysis (QC Limits: 80-120%)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 88.0% and 90.5% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr⁺⁶ concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr⁺⁶ results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)

C = Wet weight of sample (Kg)
D = % Solids/100
E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample GGMB-5_21.0 (JD13913-11) was listed as 2,940 mg/kg on the reporting form and 0.624 mg/L on the quantitation report in the raw data for a 100-fold dilution. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.624 \text{ mg/L} \times 0.1 \text{ L} \times 100}{0.00258 \text{ Kg} \times 82.3/100} = \frac{6.240}{0.0021233} = 2,938.766 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 2,940 \text{ mg/kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 2,940 mg/kg for Sample GGMB-5_21.0 was correctly reported. This was the highest detected Cr+6 concentration of the 11 detected results for the initial analysis of the 11 soil samples of this SDG, a value considerably above the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard mV solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Seven of the 11 soil samples were observed to fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The total chromium concentrations of the samples falling within the “reducing” soil conditions were all less than 360 mg/kg with Cr+6 results less than 5 mg/kg, thus considerably greater than the general Cr/Cr+6 ratio of 20:1 often exhibited in chromium containing soils (Paustenbach, et al, 1991).

Four of the samples were observed to fall on or above the phase line depicting oxidizing conditions. Three of the four samples fell above the Eh-pH phase line and contained total chromium concentrations above 1,800 mg/kg with corresponding Cr+6 results above 200 mg/kg. Sample JD13913-8 fell on the line and exhibited total chromium and Cr+6 results of 35.9 and 0.98 mg/kg, respectively.

Thus, it appears that the Eh-pH phase diagram presents Redox conditions consistent with expectations of the relationship between total chromium and Cr+6, where elevated total chromium concentrations above 500 mg/kg in oxidizing soil conditions may generate Cr+6 concentrations above the SCC of 20 mg/kg, while samples in a reducing environment generally contain less than 20 mg/kg irrespective of total chromium concentrations, but generally less than 1,000 mg/kg total chromium, based on historical observations for various PPG sites.

The results depicted on the Eh-pH diagram seem to correlate well with the observed total chromium and Cr+6 relationships.

Summary for Initial Hexavalent Chromium Analysis – SDG JD13913

Since the soluble MS spike recovery of 59.4% was below QC limits in the QC samples of QC Batch GP30170, the soil samples in this QC batch were reanalyzed within the 30-day analytical holding time. The insoluble MS recovery was within QC limits, as was the post spike recovery, as depicted in Table 5. Therefore, the Cr⁺⁶ results for the 11 samples of this QC batch in SDG JD13913 were qualified following the DV review and flagged with “NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix. Since the soil samples of this QC batch were reanalyzed, the resultant data review is presented in the section below labeled “Cr+6 Re-analyses in SDG JD13913”.

Cr+6 Re-analyses in SDG JD13913

Because the soluble MS recovery was below QC limits in the initial QC batch triggering reanalysis, the resultant data for the reanalysis of the 11 soil samples (JD13913-1R through -11R) are summarized in this section.

The QC requirements were met during the reanalysis of samples JD13913-1R through -11R in QC Batch GP30317, including the calibrations (r = 0.99986, 90.6 – 90.8% CCV Recoveries), QC blanks, and blank spike analysis (91.3% – 98.9%). The soluble MS recovery was considerably lower in the reanalysis, while the insoluble MS and post spike recoveries were within QC limits, as detailed below, while the duplicate analysis results exceeded the control limits.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The following matrix spike recoveries were observed during the reanalysis of the affected samples. However, upon reanalysis, the three spike recoveries in QC Sample JD13913-1R were less than those compared to the initial analyses, particularly in the soluble spike, as observed below in Table 6. The insoluble MS recovery in JD13913-1R was still within the 75-125% QC limits, while the post-digestion spike recovery was also within the QC limits (85-115%).

Table 6. Hexavalent Chromium Re-analysis MS Recovery Results – JD13913

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP30317 Ж	JD13913-1R	Cr ⁺⁶ , soluble	43.8 %	NJ-	Low
GP30317 Ж	JD13913-1R	Cr ⁺⁶ , insoluble	82.6 %	----	----
GP30317 Ж	JD13913-1R	Cr ⁺⁶ , post-digestion spike	89 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium Ж – The samples associated with QC Batch GP30317 consist of JD13913-1R through -11R (inclusive).					

Although the soluble MS recovery fell below 50% in the reanalysis, the criterion where DV guidelines recommend rejection of associated sample Cr+6 results (NJDEP, 2009), professional judgement was applied in qualifying the Cr+6 results of the re-analysis as estimated values, rather than possible rejection of the non-detect Cr+6 results. The Cr+6 results of the reanalysis are flagged with “NJ-”, based on data usability concepts previously discussed with Mr. Joseph

Sanguiliano of the NJDEP. Additionally, detected Cr+6 results were qualified rather than rejected, because inorganic DV guidelines do not reject detected concentration results for low spike recoveries (US EPA, 2016).

The insoluble MS recoveries were both within QC limits for the initial analysis and re-analysis, as were the post-digestion spike recoveries. The insoluble MS recovery may be a better representation of the ability of the analysis to recover Cr+6 from the soil matrix than the soluble MS recovery result, based on a great number of observations in the PPG project. Also, all but four of the 11 affected samples were observed to fall below the Eh-pH phase line demonstrating “reducing” soil conditions that do not favor oxidation of chromium to Cr+6. The samples with non-detect Cr+6 results also exhibited corresponding total chromium concentrations of less than 360 mg/kg, making it less likely that chromium would oxidize to Cr+6 in concentrations that might approach the SCC limit of 20 mg/kg. Hence, these results were qualified as estimated values and are useable based on data usability considerations, since the results of the reanalysis appear to support the Cr+6 results of the initial analysis, though they tend to be somewhat lower.

The qualified Cr+6 results of the reanalysis are presented below in Table 8 together with the results of the initial Cr+6 results.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The difference between the duplicate soil sample aliquot concentrations for Cr+6 in PPG sample JD13913-1R was 200%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010), as presented below in Table 7. The difference was also greater than 2 × CRDL, the QC limit when one or both sample concentrations are less than 5 × CRDL. The associated sample Cr+6 results were qualified as estimated values and are to be flagged with ‘*J’ because of potential variability in the analytical precision.

Table 7. Duplicate Analysis Results Outside QC Limits

QC Batch	QC Sample	Analyte	Original Result (mg/Kg)	Duplicate (mg/Kg)	Difference	DV Qualifier
GP30317 ¥	JD13913-1R	Cr+6	0.0 NJ-	1.2 NJ-	200 %RPD	*J
QC Limit is 35%RPD or < 2 × CRDL; NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result may be biased low. * – Duplicate analysis not within control limits; indeterminate bias direction. J – The reported result is an estimated value. ¥ – The samples associated with QC Batch GP30200 consist of JD13913-1R through -11R (inclusive).						

Since the duplicate analysis for Cr+6 differed by more than 35%RPD, the eleven associated PPG samples with laboratory ID numbers JD13913-1R through JD13913-11R (inclusive) are qualified as estimated values and flagged with the DV qualifier combination “*NJ-” in Table 8 due also to the low MS recovery.

Summary for Hexavalent Chromium Analysis – SDGs JD13913

The qualified soil sample results from the initial Cr+6 analysis in SDG JD13913 are presented below in Table 8 alongside those qualified results obtained from the reanalysis of samples in this SDG. Both sets of analytical Cr+6 results for samples JD13913-1 through -11 and their reanalysis

are still both qualified as estimated values (NJ-) due to a potential low bias suggested by the low soluble MS recovery results, although the insoluble MS recoveries exhibited acceptable recoveries in the initial as well as the re-analysis that were performed within the 30-day holding time. The Cr⁺⁶ concentrations determined during the re-analysis of samples in SDG JD13913 differ slightly showing lower concentrations from those of the initial analysis, but all are of similar magnitude: those Cr+6 results well below the SCC of 20 mg/kg are also below the SCC in the reanalysis, and those above the SCC are still considerably above the SCC in the reanalysis, thereby facilitating their use in project evaluations and decision making.

Table 8. Comparison of Qualified Sample Cr+6 Results in SDG JD13913 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JD13913 Result (mg/kg)	DV Qualifier	Reanalysis Result (mg/kg)	DV Qualifier
GGMB-5 1.0	JD13913-1	Cr+6	1.7	NJ-	< 0.48	*NJ-
GGMB-5 3.0	JD13913-2	Cr+6	4.1	NJ-	< 0.50	*NJ-
GGMB-5 5.0	JD13913-3	Cr+6	0.79	NJ-	< 0.45	*NJ-
GGMB-5 7.0	JD13913-4	Cr+6	0.70	NJ-	0.54	*NJ-
GGMB-5 9.0	JD13913-5	Cr+6	0.88	NJ-	< 0.46	*NJ-
GGMB-5 11.0	JD13913-6	Cr+6	4.8	NJ-	< 0.45	*NJ-
GGMB-5 13.0	JD13913-7	Cr+6	0.55	NJ-	< 0.47	*NJ-
GGMB-5 15.0	JD13913-8	Cr+6	0.98	NJ-	< 0.53	*NJ-
GGMB-5 17.0	JD13913-9	Cr+6	202	NJ-	45.4	*NJ-
GGMB-5 19.0	JD13913-10	Cr+6	1360	NJ-	1,110	*NJ-
GGMB-5 21.0	JD13913-11	Cr+6	2940	NJ-	2,620	*NJ-
<p>< –The analyte was analyzed for, but was not detected above the stated reporting limit. J – The result is an estimated value; * – Duplicate analysis not within control limits; indeterminate bias direction. NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.</p>						

Professional judgement was applied in qualifying the Cr+6 results in both analyses as estimated values (NJ-) due to a potential low bias, as suggested by the MS results tabulated above in Table 5 and Table 6. Although the soluble MS recovery was below 50% in the reanalysis of the 11 soil samples, the Cr+6 results were not rejected, but qualified as estimated values flagged with “NJ-”, based on data usability concepts previously discussed with Mr. Joseph Sanguiliano of the NJDEP. Data validation guidance rarely recommends rejection of detected sample results (USEPA, 2017) and almost all of the samples in these two QC batches exhibiting low or non-detect results for Cr+6 in the re-analysis associated with the 43.8% soluble MS recovery had corresponding total chromium results less than 360 mg/kg, most being less than 70 mg/kg, making it less likely that sample Cr+6 would approach the SCC of 20 mg/kg for the samples experiencing “reducing” soil conditions, as well as the sample falling on the Eh-pH phase line near “oxidizing” soil conditions (JD13913-8) whose total chromium concentration was less than 36 mg/kg. Additionally, the insoluble MS recoveries (87.3% and 85.3%) were within QC limits for both the initial and re-analysis and may be a better representation of the ability of the analysis to recover Cr+6 from the soil matrix than the soluble MS recovery result. The non-detect Cr+6 results in the reanalysis are supported by low (< 5 mg/kg) Cr+6 results in the initial analysis. The decision to qualify the Cr+6 results is supported by the acceptable insoluble MS recoveries and the low total chromium concentrations in the samples, as well as the acceptable post spike recoveries within QC limits for both analyses.

The sample reanalysis was performed 7 days later, within the 30-day holding time, where the Cr+6 concentrations differed only slightly upon reanalysis. The Cr+6 sample results exhibiting Cr+6 values considerably below the SCC of 20 mg/kg in the initial analysis, irrespective of the Redox state of the sample's soil environment, displayed results of similar magnitude in the reanalysis and those considerably above the SCC in the initial analysis were supported by Cr+6 results considerably above the SCC in the reanalysis, thereby enabling their use irrespective of the soluble MS recovery results.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

Although the soluble MS recovery in the reanalysis was below the 50% criterion where results are subject to rejection according to DV guideline recommendations, the results were merely qualified as estimated values rather than rejected because the results, though slightly lower in the reanalysis. The reanalysis results did confirm the results of the initial analysis in terms of magnitude, and are considered usable, as the detected results below the SCC in the initial analysis remained below the SCC and those detected above the SCC initially were also considerably above the SCC, thereby enabling project personnel to use these data in project evaluations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
*	Duplicate analysis not within control limits; indeterminate bias direction.
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

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Paustenbach, D.J., Rinehart, W.E., and P.J. Sheehan, 1991, "The Health Hazards Posed by Chromium-Contaminated Soils in Residential and Industrial Areas: Conclusions of an Expert Panel" in ***Regulatory Toxicology and Pharmacology***, 13, pp 195-222 (1991).

US EPA, CLP, 2017, ***"National Functional Guidelines for Inorganic Superfund Methods Data Review"***, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017.

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US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD13913/JD13913A

1. Were the appropriate sample preservation requirements met?..... Yes No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Samples JD13913-5A, -6A, -7A, and -8A were diluted for thallium analysis by 3x, 5x, 2x and 2x, while samples JD13913-9A and -10A were diluted 5x and 10x for the analysis of four analytes (antimony, chromium, thallium, and vanadium) due to high interfering elements. Sample JD13913-11A was diluted 50x for each of the five analytes (antimony, chromium, nickel, thallium, and vanadium), all due to high interfering elements.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

The reporting limits for antimony in Samples JD13913-10A and -11A were above the SRS of 31 mg/kg.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

Vanadium exceeded the SRS of 390 mg/kg in Samples JD13913-10A and -11A.

Cr+6 concentrations exceeded the SCC of 20 mg/kg in samples JD13913-9, -10, and -11, as well as reanalysis samples JD13913-9R, -10R, and -11R.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
 Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... Yes No

9. Were rejections noted in the non-conformance summary?..... Yes No
 Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes No

If "yes", please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds
 of concern at the site met? Yes No

12. Were the QC Summary Forms reviewed?..... Yes No

13. Internal Standards acceptable..... Yes No

14. MS/MSD acceptable..... Yes No

15. Calibration summaries acceptable..... Yes No

16. Serial dilutions acceptable..... Yes No

17. Inorganic duplicates acceptable..... Yes No

18. LCS recovery acceptable..... Yes No

19. Other QC acceptable?..... Yes No

20. Provide a brief explanation, if applicable.

Refer to DV report tables 2, 5, 6, and 7 for QC details. Qualified sample results are presented in Tables 4 and 8 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JD13987/JD13987A
Sample Dates: October 1, 2020
Analyses: Metals Analysis, EPA Method 6010D
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga. Ph.D., REP5554
Report Date: October 14, 2020

This data validation (DV) report presents the data review and result qualifications for twenty-one (21) soil samples and one (1) field blank (FB) collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey on October 1, 2020 for sample delivery group (SDG) JD13987, as well as JD13987A. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD13987A and JD13987 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 21 collected soil samples and one field blank.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JD13987-1A through JD13987-10A (inclusive), and JD13987-12A through JD13987-21A (inclusive);
- Hexavalent chromium ("*NJ-") in Samples JD13987-1 through JD13987-9 (inclusive);

No other sample results in SDG JD13987A and JD13987 required qualification, based on the acceptable remaining associated QC results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Residential Soil Remediation Standard (SRS) and Impact to Groundwater Soil Screening Level (IGWSSL) limits, whichever was more stringent, except vanadium in samples JD13987-10A and JD13987-20A, while the hexavalent chromium (Cr+6) concentrations were below the Soil Cleanup Criterion (SCC) of 20 mg/kg, except for three of the 21 soil samples in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 5 and Table 9 of this DV report.

Sample Receipt

The twenty-one (21) soil samples and one field blank collected October 1, 2020 were received intact and appropriately preserved October 1, 2020 at the SGS laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 2.3°C. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD13987A and JD13987

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
GGMB-4-1.0	JD13987-1A	10/1/2020	Soil	Metals
GGMB-4-3.0	JD13987-2A	10/1/2020	Soil	Metals
GGMB-4-5.0	JD13987-3A	10/1/2020	Soil	Metals
GGMB-4-9.0	JD13987-4A	10/1/2020	Soil	Metals
GGMB-4-11.0	JD13987-5A	10/1/2020	Soil	Metals
GGMB-4-13.0	JD13987-6A	10/1/2020	Soil	Metals
GGMB-4-15.0	JD13987-7A	10/1/2020	Soil	Metals
GGMB-4-17.0	JD13987-8A	10/1/2020	Soil	Metals
DUP02	JD13987-9A	10/1/2020	Soil	Metals
GGMB-4-19.0	JD13987-10A	10/1/2020	Soil	Metals
GGMB-4-19.0	JD13987-10AD	10/1/2020	Soil	Metals
GGMB-4-19.0	JD13987-10AS	10/1/2020	Soil	Metals
FB-02	JD13987-11A	10/1/2020	Aqueous	Metals
GGMB-4-21.0	JD13987-12A	10/1/2020	Soil	Metals
GGMB-4-32.0	JD13987-13A	10/1/2020	Soil	Metals
GGMB-3-1.0	JD13987-14A	10/1/2020	Soil	Metals
GGMB-3-3.0	JD13987-15A	10/1/2020	Soil	Metals
GGMB-3-7.0	JD13987-16A	10/1/2020	Soil	Metals
GGMB-3-9.0	JD13987-17A	10/1/2020	Soil	Metals
GGMB-3-13.0	JD13987-18A	10/1/2020	Soil	Metals
GGMB-3-15.0	JD13987-19A	10/1/2020	Soil	Metals
GGMB-3-17.0	JD13987-20A	10/1/2020	Soil	Metals
GGMB-3-19.0	JD13987-21A	10/1/2020	Soil	Metals
GGMB-3-25.0	JD13987-22A	10/1/2020	Soil	Metals
GGMB-4-1.0	JD13987-1	10/1/2020	Soil	Cr+6
GGMB-4-3.0	JD13987-2	10/1/2020	Soil	Cr+6
GGMB-4-5.0	JD13987-3	10/1/2020	Soil	Cr+6
GGMB-4-9.0	JD13987-4	10/1/2020	Soil	Cr+6
GGMB-4-11.0	JD13987-5	10/1/2020	Soil	Cr+6
GGMB-4-13.0	JD13987-6	10/1/2020	Soil	Cr+6
GGMB-4-15.0	JD13987-7	10/1/2020	Soil	Cr+6
GGMB-4-17.0	JD13987-8	10/1/2020	Soil	Cr+6
DUP02	JD13987-9	10/1/2020	Soil	Cr+6
GGMB-4-19.0	JD13987-10	10/1/2020	Soil	Cr+6
GGMB-4-19.0	JD13987-10D	10/1/2020	Soil	Cr+6
GGMB-4-19.0	JD13987-10S	10/1/2020	Soil	Cr+6
FB-02	JD13987-11	10/1/2020	Aqueous	Cr+6
GGMB-4-21.0	JD13987-12	10/1/2020	Soil	Cr+6
GGMB-4-32.0	JD13987-13	10/1/2020	Soil	Cr+6
GGMB-3-1.0	JD13987-14	10/1/2020	Soil	Cr+6
GGMB-3-3.0	JD13987-15	10/1/2020	Soil	Cr+6

GGMB-3-7.0	JD13987-16	10/1/2020	Soil	Cr+6
GGMB-3-9.0	JD13987-17	10/1/2020	Soil	Cr+6
GGMB-3-13.0	JD13987-18	10/1/2020	Soil	Cr+6
GGMB-3-15.0	JD13987-19	10/1/2020	Soil	Cr+6
GGMB-3-17.0	JD13987-20	10/1/2020	Soil	Cr+6
GGMB-3-19.0	JD13987-21	10/1/2020	Soil	Cr+6
GGMB-3-25.0	JD13987-22	10/1/2020	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010D at SGS Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JD13987A, while the data package for the hexavalent chromium analyses is numbered JD13897.

Data Review

Data, as presented in the analytical data packages SDG JD13987A and JD13987, was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) *“National Functional Guidelines for Inorganic Superfund Methods Data Review”*, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA *“ICP-AES Data Validation, SOP No. HW-3a, Revision 1”* (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JD13987A

The data validation of the metals analytical data in SDG JD13987A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|-----------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Field duplicate sample analysis |
| √ Data package completeness | √ Data qualifiers |

The 21 soil samples and one field blank were analyzed for five target EPA Method 6010D metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids, were covered by this data validation. Of the sample metals results detected in the 21 samples of SDG JD13987A, only the vanadium results in samples JD13987-10A and JD13987-20A exceeded the SRS of 390 mg/kg. No other results exhibited a concentration above the IGWSSL or SRS, whichever was more stringent.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC Batch MP23098 indicating possible matrix interference and/or sample nonhomogeneity in the soil samples analyzed in this SDG. The MS and MSD recoveries for chromium were also outside control limits, however, the spike amount was low relative to the initial sample concentration. The case narrative also stated that the RPD serial dilution result for thallium was outside control limits in QC Batch MP23098, however, the percent difference (%D) result was acceptable due to a low initial sample thallium concentration (< 50 times the instrument detection limit [IDL]). All other QC requirements for the analytes reviewed for data validation were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130%)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination) with the following exceptions.

Hence, no sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks, the continuing calibration blanks (CCBs) or field blank at the stated reporting limits.

No soil sample results warranted qualification for any associated QC blank contamination in SDG JD13987A.

ICP Interference Check Samples (QC Limits: 80-120%)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis
(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for the PPG QC batch sample JD13987-10A, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (USEPA, 2017; NJDEP, 2002).

The MS and MSD recoveries for chromium (40.2% and 164%) were also outside control limits. However, since the initial sample concentration was more than four times greater than the respective spike amount, the associated sample chromium results are not subject to qualification (USEPA, 2016), as it is likely the spikes were masked by the magnitude of the initial sample concentration.

The remaining matrix spike results fell within QC limits, including those of QC Batch GP23033 for the field blank.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP23098 Ω	JD13987-10A	Antimony	53.8 %	52.1 %	NJ-	Low

QC Limits are 75-125%;
MS – Matrix spike
MSD – Matrix spike duplicate.
NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias.
Ω – The samples associated with QC Batch MP23098 consist of JD13987-1A through -10A (inclusive) and JD13837-12A through -21A.

The antimony results in the 20 affected soil samples are flagged with “NJ-” due to a potential low bias. The antimony result in Sample JD13987-22A was not qualified as it was part of a different

QC batch whose QC results were acceptable. The 20 qualified antimony results are presented below in summary table, Table 5.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples from JD13987-10A. All %RPD values for the 5 target analytes were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 1.0 – 6.0%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging 87.8% - 101.3% for the soil sample metals analysis, and 90.0 – 98.5% for the aqueous fraction for the field blank analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative identified the serial dilution result being outside QC limits for thallium in QC Batch MP23098 and stated that the percent difference is acceptable due to a low initial sample concentration (< 50 times IDL).

The remaining serial dilution results associated with the soil samples ranged 0 – 10.0%D, values below the QC limit of 10%D criterion for data validation qualification (USEPA, 2017). No sample results required qualification for serial dilution issues.

Field Duplicate Sample Analysis (QC Limit ≤ 50%RPD)

One set of field duplicate samples were collected as part of SDG JD13987A. Field duplicate sample collection and analysis can provide a determination of sampling representativeness and precision. Gross differences between field sample duplicates can be an indication of inconsistent sampling techniques or sample matrix complexities/non-homogeneity.

The advisory data validation guidelines for field duplicate soil sample analysis vary. There is no NJDEP DV guideline for qualifying field duplicate results (NJDEP, 2002). Recently, EPA has recommended qualifying field duplicate results that differ by more than 50%RPD or > 2 × contract required quantitation limit [CRQL] (USEPA, 2016), while the Field Sampling Plan for Hudson County chromium sites lists a data quality objective (DQO) of 50%RPD for soil samples (AECOM, 2010).

The results for the analysis of the one pair of field duplicate soil samples are presented in Table 3, below. It is apparent that the results for the soil metals analytes in the field duplicate samples of GGMB-4-17.0 (JD13987-8A) were similar with results differing by less than 42%RPD or less than two times the reporting limit value (< 2 × CRQL) for all analyte pairs, thereby meeting control limits and demonstrating acceptable sample representativeness and precision.

Table 3. Comparison of Field Duplicate Soil Sample Results – SDG JD13987A

Analyte	GGMB-4-17.0 (mg/kg)	DUP02 (mg/kg)	% RPD	DV Flag
Antimony	< 12 NJ-	< 12 NJ-	0 %	-
Chromium	3,040	2,380	24.4 %	-
Nickel	261	397	41.3 %	-
Thallium	< 6.2	< 6.1	< 2 × CRQL	-

Vanadium	171	148	14.4 %	-
Total Solids	80.2 %	77.8 %	0.7 %	-
<p>< – The analyte was not detected at the stated reporting limit; NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result may be biased low. CRQL – The value representing the US EPA CLP contract required quantitation limit, often represented by the reporting limit; < 2 × CRQL – The difference between field duplicate results was less than two times the CRQL and meets QC requirements.</p>				

The field duplicate results for the field duplicate samples from GGMB-4-17.0 demonstrated acceptable sampling representativeness and precision, with field duplicate soil sample results differing by less than 50%RPD for the five target analytes. Thus, no soil sample results were qualified for sampling representativeness issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

The case narrative identified that the detection limits for various analytes were elevated in several samples due to a high interfering element.

There was one sample that required dilution that resulted in an elevated reporting limit above the SRS of 31 mg/kg for antimony. Sample JD13987-10A was diluted by a factor of 20, thereby raising the antimony reporting limit to higher than the SRS of 31 mg/kg, as detailed below in Table 4, to compensate for the interfering element presence.

Table 4. Sample Reporting Limits Affected by Sample Dilution

Sample ID	Lab ID	Analyte	Reporting Limit (mg/kg)	Dilution Factor	Adjusted Result	Remediation Standard
GGMB-4-19.0	JD13987-10A	Antimony	< 2.5	20	< 49	31
Units – mg/kg < - The analyte was analyzed for, but was not detected above the stated reporting limit.						

The interpretation of the reporting limit for antimony in sample JD13987-10A was not compromised because the vanadium concentration was above the SRS of 390 mg/kg and the sample would need to be addressed in either additional review or some type of remedial action.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JD13987A were found to be compliant with the analytical methods for the analysis of metals in the 21 soil samples and one field blank using SW-846 Method 6010D.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in the QC batch associated with the 20 soil samples of this SDG, as detailed below in Table 5. The antimony results in these samples are qualified as estimated values

(flagged “NJ-”) in the associated soil samples due to a potential low bias in the ability to recover antimony in the affected samples, as summarized below in Table 5.

Table 5. Summary of Qualified Sample Metals Results in SDG JD13987A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
GGMB-4-1.0	JD13987-1A	Antimony	< 2.3	NJ-
GGMB-4-3.0	JD13987-2A	Antimony	< 2.2	NJ-
GGMB-4-5.0	JD13987-3A	Antimony	< 2.2	NJ-
GGMB-4-9.0	JD13987-4A	Antimony	< 2.5	NJ-
GGMB-4-11.0	JD13987-5A	Antimony	< 2.4	NJ-
GGMB-4-13.0	JD13987-6A	Antimony	< 2.4	NJ-
GGMB-4-15.0	JD13987-7A	Antimony	7.9	NJ-
GGMB-4-17.0	JD13987-8A	Antimony	< 12	NJ-
DUP02	JD13987-9A	Antimony	< 12	NJ-
GGMB-4-19.0	JD13987-10A	Antimony	< 49	NJ-
GGMB-4-21.0	JD13987-12A	Antimony	< 12	NJ-
GGMB-4-32.0	JD13987-13A	Antimony	< 3.3	NJ-
GGMB-3-1.0	JD13987-14A	Antimony	< 2.3	NJ-
GGMB-3-3.0	JD13987-15A	Antimony	< 2.4	NJ-
GGMB-3-7.0	JD13987-16A	Antimony	< 2.8	NJ-
GGMB-3-9.0	JD13987-17A	Antimony	< 3.1	NJ-
GGMB-3-13.0	JD13987-18A	Antimony	< 2.3	NJ-
GGMB-3-15.0	JD13987-19A	Antimony	< 12	NJ-
GGMB-3-17.0	JD13987-20A	Antimony	< 24	NJ-
GGMB-3-19.0	JD13987-21A	Antimony	< 2.4	NJ-

Key:
 < –The analyte was analyzed for, but was not detected above the stated reporting limit.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JD13987

The analysis for hexavalent chromium (Cr⁺⁶) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in two QC batches for the 21 soil samples and one QC batch for the one field blank.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- √ Data qualifiers
- Matrix spike recoveries
- Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- Field duplicate sample analysis

Hexavalent chromium was detected in 13 of the 21 soil samples analyzed in SDG JD13987, with nine of the sample detected Cr+6 results less than 2 mg/kg, all values below the hexavalent

chromium soil cleanup criterion (SCC) of 20 mg/kg. There were, however, detected Cr+6 concentrations above the SCC of 20 mg/kg in three samples (JD13987-10, -19, and -20).

Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery of Cr+6 in QC Batch GP30200 was outside control limits indicating possible matrix interference, but the post spike was an acceptable 88%. The RPD result for the duplicate analysis was outside control limits due to possible sample nonhomogeneity. The soluble MS recovery in QC Batch GP30201 was also outside control limits, but the spike amount was low relative to the sample amount. The associated post spike recovery (91%) was also good. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% CCV Recovery)

The initial calibrations demonstrated acceptable correlation coefficients with a value of 0.99987 for both soil sample batch QC analyses and 0.99984 for the aqueous fraction, values greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered in the range of 90.2% to 91.7% for the QC batches associated with the 21 soil samples and 99.5% and 100.9% for the aqueous fraction, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg), the continuing calibration blanks, or the field blank (< 0.010 mg/L). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery of 61.7% was below the QC limits of 75-125% for QC Batch GP30200 associated with 9 soil samples, as presented below in Table 6. Thus, the hexavalent chromium results in the 9 soil samples associated with QC Batch GP30200 are subject to qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr⁺⁶ in the associated sample matrices. The insoluble MS recovery was within QC limits and the post spike also recovered within QC limits.

The soluble matrix spike recovery was also below the QC limits for QC Batch GP30201 associated with 12 soil samples, as presented below in Table 6. Although the soluble MS recovery was below 75%, the Cr+6 results in the associated 12 samples are not subject to qualification since the initial sample concentration was more than four times greater than the respective spike amount (USEPA, 2016), as it is likely the spike was masked by the magnitude of the initial sample concentration.

Table 6. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JD13987

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP30200 ¥	JD13981-1	Cr ⁺⁶ , soluble	61.7 %	NJ-	Low
GP30200 ¥	JD13981-1	Cr ⁺⁶ , insoluble	85.6 %	---	---
GP30200 ¥	JD13981-1	Cr ⁺⁶ , post-digestion spike	88 %	---	---
GP30201 £	JD13987-10	Cr ⁺⁶ , soluble	52.9 %	***	---
GP30201 £	JD13987-10	Cr ⁺⁶ , insoluble	90.1 %	---	----
GP30201 £	JD13987-10	Cr ⁺⁶ , post-digestion spike	91 %	---	---
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery					

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. *** – Sample results not subject to qualification because sample concentration is more than 4× spike amount. ¥ – The samples associated with QC Batch GP30200 consist of JD13987-1 through -9 (inclusive); £ – The samples associated with QC Batch GP30201 consist of JD13987-10, and JD13987-12 through -22 (inclusive).					

The Cr⁺⁶ results qualified for the low spike recovery in the 9 affected samples are flagged with “NJ-”, as tabulated below in Table 9. Qualification of these Cr+6 results is discussed in the Summary at the end of the Cr+6 review.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The difference between the duplicate soil sample aliquot concentrations for Cr+6 in non-client sample JD13981-1 was 148.7%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010), as presented below in Table 7. The difference was also greater than 2 × CRDL, the QC limit when one or both sample concentrations are less than 5 × CRDL. The associated sample Cr+6 results are to be qualified as estimated values and flagged with ‘*J’ because of the potential variability in the analytical precision.

Table 7. Duplicate Analysis Results Outside QC Limits

QC Batch	QC Sample	Analyte	Original Result (mg/Kg)	Duplicate (mg/Kg)	Difference	DV Qualifier
GP30200 ¥	JD13981-1	Cr+6	0.81 NJ-	5.5 NJ-	148.7 %RPD	*J

QC Limit is 35%RPD or < 2 × CRDL;
NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result may be biased low.
* – Duplicate analysis not within control limits; indeterminate bias direction.
J – The reported result is an estimated value.
¥ – The samples associated with QC Batch GP30200 consist of JD13987-1 through -9 (inclusive).

Since the duplicate analysis for Cr+6 differed by more than 35%RPD, the nine associated PPG samples with laboratory ID numbers JD13987-1 through JD13987-9 (inclusive) are qualified as estimated values and flagged with the DV qualifier combination *NJ- in Table 9, due also to the low MS recovery.

Laboratory Control Sample Analysis (QC Limits: 80-120%)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries ranging from 86.0% to 90.3% associated with the soil samples, and 101.3% for the aqueous fraction associated with the field blank, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Field Duplicate Analysis (QC Limit ≤ 50%RPD)

The results for the analysis of one set of field duplicate samples are presented in Table 8, below. The difference for the low-level concentrations observed in the field duplicate samples from sampling locations GGMB-4-17.0 differed by greater than two times the reporting level (> 5 × CRQL), the QC limit when either sample concentration is less than five times the reporting limit. The field duplicate sample results are subject to qualification as estimated values to be flagged with “J”. However, since the results are already qualified as estimated values and flagged with *NJ- for accuracy and precision issues, the two sample results were not further flagged with “J” in order to avoid a redundancy of qualifiers that do not add value to the data evaluation process.

Table 8. Comparison of Field Duplicate Soil Sample Results.

Analyte	GGMB-4-17.0 (mg/kg)	DUP02 (mg/kg)	% RPD	DV Flag
Hex.Chromium	10.3 *NJ-	1.2 *NJ-	> 2 × CRQL	(J)
<p>* – Duplicate analysis not within control limits; indeterminate bias direction. NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result may be biased low. J – The reported result is an estimated value. (J) – Sample result is subject to DV qualification, but not additionally flagged with “J” to avoid redundancy; CRQL – The value representing the US EPA CLP contract required quantitation limit, often represented by the reporting limit; > 2 × CRQL – The difference between field duplicate results was greater than two times the CRQL and exceeds QC requirements.</p>				

The field duplicate results from sampling location GGMB-4-17.0 are subject to qualification because the difference between the results was greater than two times the reporting limit, thereby exceeding the DQO for sampling representativeness and precision.

Sample Result Verification

Sample Cr⁺⁶ concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr⁺⁶ results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (Kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample GGMB-4-19.0 (JD13987-10) was listed as 410 mg/kg on the reporting form and 0.3360 mg/L on the quantitation report in the raw data for a 25-fold dilution. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.3360 \text{ mg/L} \times 0.1 \text{ L} \times 25}{0.00250 \text{ Kg} \times 82.0/100} = \frac{0.840}{0.00205} = 409.756 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 410 \text{ mg/kg}$$

After rounding to three significant figures, this verifies that the hexavalent chromium concentration of 410 mg/kg for Sample GGMB-4-19.0 was correctly reported. This was the highest detected Cr+6 concentration of the 13 detected results for the analysis of the 21 soil samples of this SDG, a value considerably above the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard mV solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Eighteen of the 21 soil samples were observed to fall below or near the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a “reducing” soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions.

Three of the samples were observed to fall above the Eh-pH phase line depicting “oxidizing” conditions. Each of these three samples exhibited total chromium concentrations above 2,000 mg/kg, as did three samples falling below the line. The three samples representing oxidizing conditions exhibited Cr+6 concentrations above the SCC of 20 mg/kg, while the three samples below the phase line with total chromium concentrations above 2,000 mg/kg exhibited Cr+6 results less than 11 mg/kg, thereby suggesting that “reducing” conditions may be effective in minimizing the oxidation of chromium to Cr+6.

The results depicted on the Eh-pH diagram seem to correlate well with the observed total chromium and Cr+6 relationships.

Summary for Hexavalent Chromium Analysis – SDGs JD13987

Since the soluble MS spike recovery of 61.7% was below QC limits in the QC samples of QC Batch GP30200, the nine soil samples in this QC batch were subject to qualification. The insoluble MS recovery was within QC limits, as was the post spike recovery, as depicted above in Table 6. Therefore, the Cr⁺⁶ results for the 9 associated samples of this QC batch in SDG JD13987 were qualified as estimated following the DV review and flagged with “*NJ-” in Table 9 due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix, as well as a potentially experiencing variability in the analytical precision as suggested by the duplicate analysis.

Although the 52.9% soluble MS recovery in QC Batch GP30201 was also below the QC limit range of 75-125%, the Cr+6 results in the 12 associated soil samples were not qualified, since the initial sample Cr+6 concentration in the spiked sample was more than four times (>4x) the spike amount

which may have masked the ability of the analysis to effectively recover Cr+6 from the sample matrix (USEPA, 2016).

The results of the Eh-pH phase diagram seem to effectively present the relationship between the observed soil sample Cr+6 concentrations and the corresponding oxidation-reduction relationship of the soil samples. Irrespective of the magnitude of the total chromium sample concentrations, for example samples with total chromium concentrations greater than 2,000 mg/kg, the samples experiencing oxidizing conditions exhibited Cr+6 results above the SCC of 20 mg/kg, while samples under “reducing” conditions exhibited Cr+6 results of ≤ 10 mg/kg.

Table 9. Summary of Qualified Cr⁺⁶ Results in JD13987

Client ID	Laboratory Sample ID	Analyte	JD13987 Result (mg/kg)	DV Qualifier
GGMB-4-1.0	JD13987-1	Cr+6	< 0.46	*NJ-
GGMB-4-3.0	JD13987-2	Cr+6	0.57	*NJ-
GGMB-4-5.0	JD13987-3	Cr+6	0.55	*NJ-
GGMB-4-9.0	JD13987-4	Cr+6	1.1	*NJ-
GGMB-4-11.0	JD13987-5	Cr+6	0.65	*NJ-
GGMB-4-13.0	JD13987-6	Cr+6	1.8	*NJ-
GGMB-4-15.0	JD13987-7	Cr+6	< 0.48	*NJ-
GGMB-4-17.0	JD13987-8	Cr+6	10.3	*NJ-
DUP02	JD13987-9	Cr+6	1.2	*NJ-
<p>< –The analyte was analyzed for, but was not detected above the stated reporting limit. J – The reported result is an estimated value. * – Duplicate analysis not within control limits; indeterminate bias direction. NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result may be biased low.</p>				

The qualified soil sample results from the QC Batch GP30200 analysis in SDG JD13987 are presented above in Table 9. The analytical Cr⁺⁶ results for samples JD13987-1 through -9 are qualified as estimated values (*NJ-) due to a potential low bias.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
*	Duplicate analysis not within control limits; indeterminate bias direction.
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

Kahn, Lloyd, 1988, ***Determination of Total Organic Carbon in Sediment (Lloyd Kahn Method)***, July 27, 1988: Edison, NJ, U.S. Environmental Protection Agency, Region II, 5 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

US EPA, CLP, 2017, ***“National Functional Guidelines for Inorganic Superfund Methods Data Review”***, OSWER Publication 9355.0-135, EPA-540-R-2017-001, January 2017.

US EPA, 2016, ***ICP-AES Data Validation, SOP HW-3a, Revision 1***, December 2016.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD13987/JD13987A

1. Were the appropriate sample preservation requirements met?..... Yes No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.
Samples JD13987-6A, -7A, -16A, -17A, -18A, -21A, and -22A were diluted for thallium analysis, while samples JD13987-8A, -9A, -10A, -12A, -19A, and -20A were diluted for the analysis of four analytes (antimony, chromium, thallium, and vanadium) due to high interfering elements.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.
The reporting limit for antimony in Sample JD13987-10A was above the SRS of 31 mg/kg.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.
The concentrations of vanadium in Samples JD13987-10A and JD13987-20A were above the SRS of 390 mg/kg. Cr+6 exceeded the SCC of 20 mg/kg in samples JD13987-10, -19, and -20.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... Yes No
9. Were rejections noted in the non-conformance summary?..... Yes No
Provide a brief explanation.
- Not applicable**
10. Were rejected data used?..... Yes No
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes No
12. Were the QC Summary Forms reviewed?..... Yes No
13. Internal Standards acceptable..... Yes No
14. MS/MSD acceptable..... Yes No
15. Calibration summaries acceptable..... Yes No
16. Serial dilutions acceptable..... Yes No
17. Inorganic duplicates acceptable..... Yes No
18. LCS recovery acceptable..... Yes No
19. Other QC acceptable?..... Yes No
20. Provide a brief explanation, if applicable.

The field duplicate sample results for location GGMB-4-17.0 (JD13987-8 and -9) differed by more than the QC limit for Cr+6 (Table 8). Sample Cr+6 results were not additionally flagged with “J”, since results are already qualified as estimated values and flagged with *NJ- in Table 9.

Refer to DV report tables 2, 3, 6, 7, and 8 for QC details. Qualified sample results are presented in Tables 5 and 9 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JD14204/JD14204A
Sample Dates: October 5 – 6, 2020
Analyses: Metals Analysis, EPA Method 6010D
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: October 27, 2020

This data validation (DV) report presents the data review and result qualifications for five (5) soil samples and one (1) field blank (FB) collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey on October 5-6, 2020 for sample delivery group (SDG) JD14204, as well as JD14204A. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD14204A and JD14204 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 5 collected soil samples and one field blank.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JD14204-1A through JD14204-5A (inclusive);
- Hexavalent chromium ("NR") in Samples JD14204-1 through JD14204-5 (inclusive);
- Hexavalent chromium ("NR") in reanalysis samples JD14204-1R through JD14204-5R (inclusive)

No other sample results in SDG JD14204A and JD14204 required qualification, based on the acceptable remaining associated QC results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Residential Soil Remediation Standard (SRS) and Impact to Groundwater Soil Screening Level (IGWSSL) limits, whichever was more stringent, except for vanadium in Sample JD14204-4A, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) of 20 mg/kg in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 3 and Table 6 of this DV report.

Sample Receipt

The five (5) soil samples and one field blank collected October 5-6, 2020 were received intact and appropriately preserved October 6, 2020 at the SGS laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 3.4°C. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD14204A and JD14204

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
GGMB-1W-0.0	JD14204-1A	10/5/2020	Soil	Metals
GGMB-1W-2.0	JD14204-2A	10/5/2020	Soil	Metals
GGMB-1W-4.0	JD14204-3A	10/5/2020	Soil	Metals
GGMB-1W-7.0	JD14204-4A	10/5/2020	Soil	Metals
GGMB-2W-2.0	JD14204-5A	10/5/2020	Soil	Metals
FB-WG1	JD14204-6A	10/6/2020	Aqueous	Metals
GGMB-1W-0.0	JD14204-1	10/5/2020	Soil	Cr+6
GGMB-1W-2.0	JD14204-2	10/5/2020	Soil	Cr+6
GGMB-1W-4.0	JD14204-3	10/5/2020	Soil	Cr+6
GGMB-1W-7.0	JD14204-4	10/5/2020	Soil	Cr+6
GGMB-2W-2.0	JD14204-5	10/5/2020	Soil	Cr+6
FB-WG1	JD14204-6	10/6/2020	Aqueous	Cr+6
				Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010D at SGS Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JD14204A, while the data package for the hexavalent chromium analyses is numbered JD14204.

Data Review

Data, as presented in the analytical data packages SDG JD14202A and JD14204 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Superfund Methods Data Review*”, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA “*ICP-AES Data Validation, SOP No. HW-3a, Revision 1*” (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JD14204A

The data validation of the metals analytical data in SDG JD14204A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The five soil samples and one field blank were analyzed for the five target EPA Method 6010D metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids, were covered by this data validation. Of the sample metals results detected in the 5 samples of SDG JD14204, no results exhibited a concentration above the IGWSSL or SRS, whichever was more stringent, except for the vanadium concentration of 1,450 mg/kg in Sample JD14204-4A.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC Batch MP23179 indicating possible matrix interference and/or sample nonhomogeneity in the soil samples analyzed in this SDG. Samples JD14204-1A, -4A, and -5A were diluted for antimony, thallium, and vanadium analysis, while sample JD14204-2A was diluted for the analysis of thallium, each due to high interfering elements. The case narrative also stated that the MSD RPD result for chromium was outside control limits in QC Batch MP23179 and the high RPD may be due to possible sample

nonhomogeneity. The case narrative also stated that the RPD serial dilution result for thallium was outside control limits in QC Batch MP23179, however, the percent difference (%D) result was acceptable due to a low initial sample thallium concentration (< 50 times the instrument detection limit [IDL]). All other QC requirements for the analytes reviewed for data validation were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130%)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Hence, no sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks, the continuing calibration blanks (CCBs) or field blank at the stated reporting limit (RL) or contract required detection limit (CRDL). No soil sample results warranted qualification for any associated QC blank contamination in SDG JD14204A.

ICP Interference Check Samples (QC Limits: 80-120%)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis
(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were below the QC limits of 75 - 125% for the PPG QC batch sample JD14204-5A, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (USEPA, 2017; NJDEP, 2002). The remaining matrix spike results fell within QC limits, including those of QC Batch GP23192 for the field blank.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP23179 Ω	JD14204-5A	Antimony	54.2 %	50.7 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a						

potential low bias.

Ω – The samples associated with QC Batch MP23179 consist of JD14204-1A through -5A (inclusive).

The case narrative stated that the RPD result for chromium between the spiked duplicate samples (21.5%) was outside control limits. However, applying professional judgement in the DV review, the associated sample chromium results were not qualified, since DV guidelines (USEPA, 2017) allow for use of a less restrictive project data quality objective (DQO) such as 35% or 2×CRQL to be assessed against duplicate soil samples, as identified in the section title above.

The antimony results in the five affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples from JD14204-5A. All %RPD values for the 5 target analytes were below the laboratory QC limit of 20%RPD, except for the 21.4% results for chromium, but which was below the project QC limit of 35%RPD for soil samples, with values ranging 0.3 – 21.4%RPD for soil samples with no results requiring qualification, and 0.5 – 1.1% for QC Batch MP23192 associated with the field blank. The duplicate analyses demonstrated acceptable analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging 93.6% - 96.0% for the soil sample metals analysis, and 94.5 – 97.0% for the aqueous fraction for the field blank analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative identified the serial dilution result being outside QC limits for thallium in QC Batch MP23179 and stated that the percent difference is acceptable due to low initial sample concentration (< 50 times IDL).

The remaining serial dilution results associated with the soil samples ranged from 0 to 7.9%D, values below the QC limit of 10%D criterion for data validation qualification (USEPA, 2017). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

Although various analytes in several samples were diluted for high interfering elements, no samples were diluted to the extent that the reporting limit exceeded the respective IGWSSL or SRS limit values.

Hence, all reporting limits were below the respective project IGWSSL and SRS limit values.

Summary of Qualified Metals Results in JD14204A

The soil sample analytical results for the samples of SDG JD14204A were found to be compliant with the analytical methods for the analysis of metals in the 5 soil samples and one field blank using SW-846 Method 6010D.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in the QC batch associated with the 5 soil samples of this SDG, as detailed below in Table 3. The antimony results in these samples are qualified as estimated values (flagged "NJ-") in the associated soil samples due to a potential low bias in the ability to recover antimony in the affected samples, as listed below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JD14204A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
GGMB-1W-0.0	JD14204-1A	Antimony	< 7.2	NJ-
GGMB-1W-2.0	JD14204-2A	Antimony	< 2.5	NJ-
GGMB-1W-4.0	JD14204-3A	Antimony	< 2.3	NJ-
GGMB-1W-7.0	JD14204-4A	Antimony	< 17	NJ-
GGMB-2W-2.0	JD14204-5A	Antimony	< 12	NJ-

Key:
< –The analyte was analyzed for, but was not detected above the stated reporting limit.
NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JD14204

The analysis for hexavalent chromium (Cr⁺⁶) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the 5 soil samples and one QC batch for the field blank. The soil samples were re-analyzed in an additional QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | √ Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was not detected in any of the five soil samples initially analyzed in SDG JD14204, nor any of the samples in the reanalysis effort. Hence, all sample Cr⁺⁶ results are less than the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble and insoluble matrix spike recoveries in QC Batch

GP30267 were outside control limits, along with low post spike and pH-adjusted post spike recoveries, thereby suggesting that the recoveries indicate possible matrix interference. The soluble and insoluble matrix spike recoveries in reanalysis QC Batch GP30389 were also outside control limits, along with low post spike and pH-adjusted post spike recoveries. There was good agreement between the sample and 1:5 dilution in QC Batch GP30267 and GP30389. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% CCV Recovery)

The initial calibrations demonstrated acceptable correlation coefficients with a value of 0.99996 for the soil sample analysis and 0.99971 for the aqueous fraction, values greater than the calibration requirement for linearity of 0.995, as was the 0.99985 correlation coefficient in the reanalysis. The calibration check standard recoveries were 90.8% for the QC batch associated with the 5 soil samples and ranged from 100.4% to 101.8% for the aqueous fraction, all meeting the continuing calibration QC requirement of 90-110%. The calibration check standard recoveries were 90.4% - 90.6% for the QC batch associated with the reanalysis of the 5 soil samples.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg), the continuing calibration blanks, or the field blank (< 0.010 mg/L). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble and insoluble matrix spike recoveries were below the QC limits of 75-125% for QC Batch GP30267 associated with 5 soil samples, with recoveries of 0% and 0.0%, respectively, as presented below in Table 4. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP30267 are subject to rejection based on the results of the soluble MS and insoluble MS recoveries below 50%, as recommended in the DV guidelines for Cr+6 analysis (NJDEP, 2009) and the perceived inability to recover Cr+6 in the associated sample matrices. The post spike and pH-adjusted post spikes also recovered below QC limits such that the results are subject to qualification.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JD14204

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP30267 ¥	JD14204-1	Cr ⁺⁶ , soluble	0.0 %	NR	Low
GP30267 ¥	JD14204-1	Cr ⁺⁶ , insoluble	0.0 %	NR	Low
GP30267 ¥	JD14204-1	Cr ⁺⁶ , post-digestion spike	17.5 %	---	Low
GP30267 ¥	JD14204-1	Cr ⁺⁶ , pH-adjusted post spike	20.9 %	---	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium N – The matrix spike sample recovery in the associated QC sample is outside QC limits R – The result is rejected because the MS recovery in the associated QC sample is below 50%; ¥ – The samples associated with QC Batch GP30267 consist of JD14204-1 through -5 (inclusive).					

The Cr⁺⁶ results associated with MS recoveries below 50% are rejected and flagged with “NR”, as tabulated below in Table 6, together with the rejected results from the re-analysis of this QC batch.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JD14204-1. The difference between the duplicate soil sample aliquots for Cr+6 in soil this sample (GGMB-1W-0.0) was listed as 0.0%RPD, a value below the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010). The RPD value for the aqueous QC Batch GN12095 associated with the field blank was also 0.0%. The %RPD values for redox potential (9.9%RPD) and pH (0.2%RPD) displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was within the QC limit for soil samples, the associated sample results are not subject to qualification and represent acceptable analytical precision.

Laboratory Control Sample Analysis (QC Limits: 80-120%)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 92.3% and 95.3% associated with the soil samples, and 103.3% for the aqueous fraction associated with the field blanks, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr⁺⁶ results were qualified for serial dilution analysis results. Serial dilution is not a requirement of the analytical method, although it appears that a serial dilution analysis was performed in the analytical sequence. A note on the MS Results Summary page indicated that there was good agreement between the sample and the 1:5 dilution in the analysis QC batch.

Sample Result Verification

Sample Cr⁺⁶ concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr⁺⁶ results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (Kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The non-detect hexavalent chromium concentration for Sample GGMB-1W-4.0 (JD14204-3) was listed as < 0.48 mg/kg on the reporting form and 0.0063 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0063 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00250 \text{ Kg} \times 83.7/100} = \frac{0.00063}{0.0020925} = 0.30108 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 0.96 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the non-detect hexavalent chromium result of < 0.48 mg/kg for Sample GGMB-1W-4.0 was correctly reported. This was one of the five non-detect Cr+6 concentrations for the initial analysis of the 5 soil samples of this SDG.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard mV solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Each of the 5 soil samples were observed to fall considerably below or just below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The total chromium concentrations of the samples falling below the Eh-pH phase line representing “reducing” soil conditions ranged from 50.2 mg/kg to 5,710 mg/kg, a rather wide range.

It is possible that the reducing soil environment contributed significantly to the poor MS recoveries observed in the initial soil analysis, conditions which do not favor oxidation of chromium to Cr+6. Despite the poor MS recoveries, it is possible that the reducing environment conditions exhibited by the Eh-pH phase diagram contribute significantly to the observed low or non-detect Cr+6 results and may reflect somewhat representative results, due to the reducing soil conditions.

Summary for Initial Hexavalent Chromium Analysis – SDG JD14204

Since the soluble MS spike recovery of 0.0% was below QC limits in the QC sample of QC Batch GP30267, the soil samples in this QC batch required reanalysis. The 0.0% insoluble MS recovery was also below 50%, while the post spike and pH-adjusted post spike recoveries were below the respective QC limits, as depicted in Table 4. Therefore, the Cr⁶ results for the 5 samples of this QC batch in SDG JD14204 were rejected following the DV review and flagged with “NR” due to a potential inability to recover hexavalent chromium from the soil sample matrix. Consequently, the soil samples of this QC batch were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analyses in SDG JD14204”.

Cr+6 Re-analyses in SDG JD14204

Because the soluble MS recovery was below QC limits in the initial QC batch triggering reanalysis, the resultant data for the reanalysis of the 5 soil samples (JD14204-1R through -5R) are summarized in this section.

The QC requirements were met during the reanalysis of samples JD14204-1R through -5R in QC Batch GP30389, including the calibrations ($r = 0.99985$, 90.4% - 90.6% CCV Recoveries), QC blanks, duplicate analysis (0.0 %), and blank spike analysis (93.3% – 100.6%). The 0.0% soluble MS recovery in the reanalysis was similar to the 0% recovery in the initial analysis, as were the 0% insoluble MS recovery, post spike (11%) and pH-adjusted post spike (30%) recoveries, but still recovering below the respective QC limits, as detailed below in Table 5.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble MS and insoluble MS recoveries were still below 50% in the reanalysis, thereby confirming the decision to reject the Cr+6 sample results in both the initial and reanalysis. The low recoveries of the post-digestion spike and pH-adjusted post spike analysis support the observations of the soluble and insoluble MS recoveries.

The following matrix spike recoveries were observed during the reanalysis of the affected samples (Table 5).

Table 5. Hexavalent Chromium Re-analysis MS Recovery Results – JD14204

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP30389 X	JD14204-1R	Cr ⁺⁶ , soluble	0.0 %	NR	Low
GP30389 X	JD14204-1R	Cr ⁺⁶ , insoluble	0.0 %	NR	Low
GP30389 X	JD14204-1R	Cr ⁺⁶ , post-digestion spike	11 %	----	Low
GP30389 X	JD14204-1R	Cr ⁺⁶ , pH-adjusted post spike	30 %	---	Low
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium X – The samples associated with QC Batch GP30389 consist of JD14204-1R through -5R (inclusive).					

Since the soluble and insoluble MS recoveries in QC Batch GP30389 are still below 50%, the Cr⁺⁶ results for the samples in this QC batch are also subject to rejection to be flagged with “NR” for a potential inability to recover Cr⁺⁶ in this QC batch. The qualified Cr+6 results of the reanalysis are presented below in Table 6 together with the results of the initial Cr+6 analysis.

Summary for Hexavalent Chromium Analysis – SDGs JD14204

The qualified (rejected) soil sample results from the initial Cr+6 analysis in SDG JD14204 are presented below in Table 6 alongside those qualified results obtained from the reanalysis of samples in this SDG. Both sets of analytical Cr⁺⁶ results for samples JD14204-1 through -5 (inclusive) and their reanalysis are still both rejected (“NR”) due to the very low spike recoveries and a potential low bias, as recommended by NJDEP DV guidelines (NJDEP, 2009). The Cr⁺⁶ concentrations determined during the re-analysis of samples in SDG JD14204, that were performed 7 days later within the 30-day holding time, are essentially the same as those of the initial analysis, being all non-detect results, still well below the SCC of 20 mg/kg.

Table 6. Comparison of Qualified Cr⁺⁶ Results in JD14204 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JD14204 Result (mg/kg)	DV Qualifier	Reanalysis Results (mg/kg)	DV Qualifier
GGMB-1W-0.0	JD14204-1	Cr+6	< 0.49	NR	< 0.50	NR
GGMB-1W-2.0	JD14204-2	Cr+6	< 0.47	NR	< 0.46	NR
GGMB-1W-4.0	JD14204-3	Cr+6	< 0.48	NR	< 0.47	NR
GGMB-1W-7.0	JD14204-4	Cr+6	< 0.66	NR	< 0.68	NR
GGMB-2W-2.0	JD14204-5	Cr+6	< 0.48	NR	< 0.49	NR
< –The analyte was analyzed for, but was not detected above the stated reporting limit. N – The matrix spike sample recovery in the associated QC sample is outside QC limits R – The result is rejected because the MS recovery in the associated QC sample is below 50%; NR – The matrix spike sample recovery in the associated QC sample is below the 50% QC limit recommending rejection of the result.						

Although the Cr+6 results were rejected in all 5 soil samples in the initial analysis, as well as the reanalysis in accordance with DV guidance (NJDEP, 2009), the guidance also suggests that the Eh-pH results can be referred to for data usability.

Despite the poor MS recoveries, it is possible that the reducing environment conditions exhibited by the Eh-pH phase diagram contribute significantly to the observed non-detect Cr+6 results and may actually reflect somewhat representative results because of the reducing soil conditions that do not favor oxidation of chromium to Cr+6.

Additionally, the results of the reanalysis were extremely similar to the non-detect results of the initial analysis, and seemingly irrespective of the total chromium concentration.

These results might be considered for site use, but with extreme caution, in conjunction with other site information.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
R	The result is rejected because the MS recovery in the associated QC sample is below 50%;

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

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New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, CLP, 2017, ***“National Functional Guidelines for Inorganic Superfund Methods Data Review”***, OSWER Publication 9355.0-135, EPA-540-R-2017-001, January 2017.

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US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

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ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD14204/JD14204A

1. Were the appropriate sample preservation requirements met?..... Yes No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Samples JD14204-1A, -4A, and -5A were diluted for antimony, thallium and vanadium analysis, while Sample JD14204-2A was diluted for thallium. These samples were diluted due to high interfering elements.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

The concentration of vanadium in Sample JD14204-4A exceeded the SRS of 390 mg/kg.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.
10. Were rejected data used?..... **Yes** No
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
20. Provide a brief explanation, if applicable.

Refer to DV report tables 2, 4, and 5 for QC details. Qualified sample results are presented in Tables 3 and 6 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JD16567/JD16567A
Sample Dates: November 19, 2020
Analyses: Metals Analysis, EPA Method 6010D
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga. Ph.D., REP5554
Report Date: January 26, 2021

This data validation (DV) report presents the data review and result qualifications for four (4) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey on November 19, 2020 for sample delivery group (SDG) JD16567, as well as JD16567A. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD16567A and JD16567 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 4 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JD16567-1A through JD16567-4A (inclusive);
- Hexavalent chromium ("NJ-") in Samples JD16567-1 through JD16567-4 (inclusive);

No other sample results in SDG JD16567A and JD16567 required qualification, based on the acceptable remaining associated QC results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Residential Soil Remediation Standard (SRS) and Impact to Groundwater Soil Screening Level (IGWSSL) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC). A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 3 and Table 5 of this DV report.

Sample Receipt

The four (4) post-excavation soil samples collected November 19, 2020 received intact and appropriately preserved November 19, 2020 at the SGS laboratory in Dayton, NJ with acceptable

sampling cooler temperatures with a maximum corrected temperature of 2.9°C. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD16567A and JD16567

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
FNC-2A	JD16567-1A	11/19/2020	Soil	Metals
FNC-2B	JD16567-2A	11/19/2020	Soil	Metals
FNC-3A	JD16567-3A	11/19/2020	Soil	Metals
FNC-3B	JD16567-4A	11/19/2020	Soil	Metals
FNC-2A	JD16567-1	11/19/2020	Soil	Cr+6
FNC-2B	JD16567-2	11/19/2020	Soil	Cr+6
FNC-3A	JD16567-3	11/19/2020	Soil	Cr+6
FNC-3B	JD16567-4	11/19/2020	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010D at SGS Laboratories in Dayton, NJ, as well as percent total solids.				
Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JD16567A, while the data package for the hexavalent chromium analyses is numbered JD16567.

Data Review

Data, as presented in the analytical data packages SDG JD16567A and JD16567 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Superfund Methods Data Review*”, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA “*ICP-AES Data Validation, SOP No. HW-3a, Revision 1*” (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical

Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JD16567A

The data validation of the metals analytical data in SDG JD16567A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The four soil samples were analyzed for the five target EPA Method 6010D metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids, were covered by this data validation. Of the sample metals results detected in the 4 soil samples of SDG JD16567A, no results in the four samples exhibited a concentration above the IGWSSL or SRS, whichever was more stringent.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC Batch MP23905 indicating possible matrix interference and/or sample nonhomogeneity in the soil samples analyzed in this SDG. No samples were diluted for the metals analysis. All other QC requirements for the analytes reviewed for data validation were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130%)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard),

with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Hence, no sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks and the continuing calibration blanks (CCBs) at the stated reporting limit (RL) or contract required detection limit (CRDL).

No soil sample results warranted qualification for any associated QC blank contamination in SDG JD16567A.

ICP Interference Check Samples (QC Limits: 80-120%)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis
(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries for antimony were below the QC limits of 75 - 125% for the non-client QC batch sample JD16515-15, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (USEPA, 2017; NJDEP, 2002). The remaining matrix spike results fell within QC limits in Batch MP23905.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP23905 Ω	JD16515-15	Antimony	57.1 %	54.6 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias. Ω – The samples associated with QC Batch MP23905 consist of JD16567-1A through -4A (inclusive).						

The antimony results in the four affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples from non-client sample JD16515-15. All %RPD values for the 5 target analytes were below the laboratory QC limit

of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 4.4 – 8.6%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated acceptable analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging 90.8% - 100.5% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The serial dilution results associated with the soil samples ranged 0 – 5.0%D, values below the QC limit of 10%D criterion for data validation qualification (USEPA, 2017). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Hence, all reporting limits were below the respective project IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JD16567A were found to be compliant with the analytical methods for the analysis of metals in the four soil samples using SW-846 Method 6010D.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in the QC batch associated with the 4 soil samples of this SDG, as detailed above in Table 2. The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias in the ability to recover antimony in the affected samples, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JD16567A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
FNC-2A	JD16567-1A	Antimony	< 2.6	NJ-
FNC-2B	JD16567-2A	Antimony	< 2.3	NJ-
FNC-3A	JD16567-3A	Antimony	< 2.3	NJ-
FNC-3B	JD16567-4A	Antimony	< 2.3	NJ-

Key:
 < –The analyte was analyzed for, but was not detected above the stated reporting limit.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JD16567

The analysis for hexavalent chromium (Cr⁺⁶) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the four (4) soil samples.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was detected in three of the 4 post-excavation soil samples analyzed initially in SDG JD16567. No soil sample Cr+6 results in the samples of SDG JD16567 exceeded the soil cleanup criterion (SCC) of 20 mg/kg, with results being less than 3 mg/kg when detected.

Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery in QC Batch GP30980 was outside control limits, while the insoluble MS recovery and post spike recoveries were within control limits. The RPD value for the duplicate analysis in QC Batch GP30980 was outside control limits, but was acceptable due to low duplicate and sample concentrations. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% CCV Recovery)

The initial calibration demonstrated an acceptable correlation coefficient with a value of 0.99992 for the soil sample analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standard recoveries were all 92.6% for the QC batch associated with the 4 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks. Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP30980 associated with 4 soil samples, as presented below in Table 4. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP30980 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr⁺⁶ in the associated sample matrices. The insoluble MS recovery was within QC limits and the post spike also recovered within QC limits.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JD16567

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP30980 ¥	JD16644-1	Cr ⁺⁶ , soluble	35.1 %	NJ-/NR	Low
GP30980 ¥	JD16644-1	Cr ⁺⁶ , insoluble	94.6 %	----	----
GP30980 ¥	JD16644-1	Cr ⁺⁶ , post-digestion spike	93.1 %	----	----

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
MS – Matrix spike
Cr⁺⁶ – Hexavalent chromium
NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
NR – The matrix spike sample recovery in the associated QC sample is below the 50% QC limit recommending rejection of the non-detect result.
¥ – The samples associated with QC Batch GP30980 consist of JD16567-1 through -4 (inclusive).

The matrix spike (MS) recovery of 94.6% for the insoluble spike was acceptable, while the soluble MS recovery (35.1%) for the spiked sample JD16644-1 from soil sampling location FNC-4A in PPG SDG JD16644 was below the lower QC limit of 75%, as well as below the 50% criterion where NJDEP DV guidelines (NJDEP, 2009) recommend rejection of associated Cr+6 results. However, professional judgement was applied in not rejecting the results in the associated samples for various reasons discussed below in the Summary section at the end of the Cr+6 review.

Thus, the Cr⁺⁶ results for the 4 samples of this QC batch in SDG JD16567 were qualified following the DV review and flagged with “NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix. The qualified Cr+6 results are presented below in Table 5.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JD16644-1. The difference between the duplicate soil sample aliquots for Cr+6 in soil this PPG sample (FNC-4A from JD16644) was listed as 200.0%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010). However, the difference between the sample (0.45 mg/kg) and its duplicate (0.0 mg/kg) was a value less than two times the reporting limit of < 0.48 mg/kg, the QC limit when either concentration is less than five times the reporting limit. The %RPD values for redox potential (9.2%RPD) and pH (1.2%RPD) displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was within the QC limit of “< 2 × 0.48” mg/kg for soil samples, the associated sample results in the initial analysis are not subject to qualification and represent acceptable analytical precision.

Laboratory Control Sample Analysis (QC Limits: 80-120%)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 85.0% and 93.0% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr⁺⁶ concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr⁺⁶ results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)
 C = Wet weight of sample (Kg)
 D = % Solids/100
 E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample FNC-3A (JD16567-3) was listed as 2.8 mg/kg on the reporting form and 0.059 mg/L on the quantitation report in the raw data for an undiluted sample. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.059 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00248 \text{ Kg} \times 85.2/100} = \frac{0.0059}{0.0021130} = 2.7922 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 2.8 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 2.8 mg/kg for Sample FNC-3A was correctly reported. This was the highest detected Cr+6 concentration of the three detected results for the initial analysis of the 4 soil samples of this SDG, a value considerably below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard mV solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Each of the four soil samples were observed to fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The total chromium concentrations of the samples falling within the “reducing” soil conditions were all less than 40 mg/kg with Cr+6 results less than 3 mg/kg. This is not inconsistent with a study of chromium contaminated soils in New Jersey demonstrated that the general ratio of chromium to Cr+6 was typically a ratio of 20:1 (Paustenbach, et al, 1991).

Thus, it appears that the Eh-pH phase diagram presents Redox conditions consistent with expectations of the relationship between total chromium and Cr+6, where total chromium

concentrations below 40 mg/kg in reducing soil conditions are highly unlikely to oxidize to Cr+6 concentrations that could approach the SCC of 20 mg/kg.

Summary for Hexavalent Chromium Analysis – SDG JD16567

The matrix spike (MS) recovery of 94.6% for the insoluble spike was acceptable, while the soluble MS recovery (35.1%) for the spiked sample JD16644-1 from soil sampling location FNC-4A in PPG SDG JD16644 was below the lower QC limit of 75%, as well as below the 50% criterion where NJDEP DV guidelines (NJDEP, 2009) recommend rejection of associated Cr+6 results. However, professional judgement was applied in not rejecting the results in the associated samples for various reasons.

Rarely do DV guidelines recommend rejection of detected concentration results, but rather recommend qualification as estimated values. In DV guidance for inorganic analytes (USEPA, 2017), rejection of results is recommended when MS recoveries fall below 30% for non-detect results, but recommend qualification as estimated values (flag with “J”) for detected concentrations. Secondly, the insoluble recovery was well within QC limits and may be a better representation of the ability to recover Cr+6 from a soil matrix, rather than the soluble MS recovery result. Thirdly, because the total chromium result in the associated sample exhibiting a non-detect Cr+6 result (JD16567-2) was 16.1 mg/kg, it is extremely unlikely that Cr+6 results would approach the SCC of 20 mg/Kg, since all samples of this SDG were represented by “reducing” soil conditions which are unfavorable for oxidation of chromium to Cr+6. Additionally, the post-digestion spike was also acceptable (94.8%).

Although the soluble MS recovery in the reanalysis was below the 50% criterion where results are subject to rejection according to DV guideline recommendations, based on professional judgement, the results were merely qualified as estimated values and flagged with “NJ-” rather than rejected. The decision not to reject the soil sample Cr+6 results was based on the following: the reducing nature of the soil conditions for all four samples, the very low total chromium concentrations in each of the four samples, and the favorable insoluble MS recovery, a result that is more likely representative of the analytical accuracy for a soil matrix.

The qualified soil sample results from the Cr+6 analysis in SDG JD16567 are presented below in Table 5. The analytical Cr⁺⁶ results for samples JD16567-1 through -4 are qualified as estimated values (NJ-) due to a potential low bias suggested by the low soluble MS recovery results, although the insoluble MS analysis exhibited a very good recovery. The Cr⁺⁶ concentrations determined during the analysis of samples in SDG JD16567 are of similar low magnitude and well below the SCC of 20 mg/kg.

Table 5. Qualified Sample Cr+6 Results in SDG JD16567

Client ID	Laboratory Sample ID	Analyte	Result (mg/kg)	DV Qualifier
FNC-2A	JD16567-1	Cr+6	0.76	NJ-
FNC-2B	JD16567-2	Cr+6	< 0.46	NJ-
FNC-3A	JD16567-3	Cr+6	2.8	NJ-
FNC-3B	JD16567-4	Cr+6	0.52	NJ-
< –The analyte was analyzed for, but was not detected above the stated reporting limit. NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.				

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

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New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

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New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

Paustenbach, D.J., Rinehart, W.E., and P.J. Sheehan, 1991, "The Health Hazards Posed by Chromium-Contaminated Soils in Residential and Industrial Areas: Conclusions of an Expert Panel" in ***Regulatory Toxicology and Pharmacology***, 13, pp 195-222 (1991).

US EPA, CLP, 2017, ***"National Functional Guidelines for Inorganic Superfund Methods Data Review"***, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017.

US EPA, 2016, ***ICP-AES Data Validation, SOP HW-3a, Revision 1***, December 2016.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD16567/JD16567A

1. Were the appropriate sample preservation requirements met?..... Yes No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.
- Not applicable**
10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
20. Provide a brief explanation, if applicable.

Refer to DV report tables 2 and 4 for QC details. Qualified sample results are presented in Tables 3 and 5 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JD16644/JD16644A
Sample Dates: November 20, 2020
Analyses: Metals Analysis, EPA Method 6010D
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga. Ph.D., REP5554
Report Date: January 26, 2021

This data validation (DV) report presents the data review and result qualifications for two (2) post-excavation soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey on November 20, 2020 for sample delivery group (SDG) JD16644, as well as JD16644A. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD16644A and JD16644 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 2 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Hexavalent chromium ("NJ-") in Samples JD16644-1 and JD16644-2.

No other sample results in SDG JD16644A and JD16644 required qualification, based on the acceptable remaining associated QC results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Residential Soil Remediation Standard (SRS) and Impact to Groundwater Soil Screening Level (IGWSSL) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC). A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 3 of this DV report.

Sample Receipt

The two (2) post-excavation soil samples collected November 20, 2020 received intact and appropriately preserved November 20, 2020 at the SGS laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 1.6°C. The field sample

identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD16644A and JD16644

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
FNC-4A	JD16644-1A	11/20/2020	Soil	Metals
FNC-4B	JD16644-2A	11/20/2020	Soil	Metals
FNC-4A	JD16644-1	11/20/2020	Soil	Cr+6
FNC-4B	JD16644-2	11/20/2020	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010D at SGS Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JD16644A, while the data package for the hexavalent chromium analyses is numbered JD16644.

Please note that the data package also contained data for two additional samples (FNC-5A and FNC-5B) that were not subject to data validation because the sample locations were resampled and will not be addressed in this DV report.

Data Review

Data, as presented in the analytical data packages SDG JD16644A and JD16644 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16* (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) *“National Functional Guidelines for Inorganic Superfund Methods Data Review”*, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA *“ICP-AES Data Validation, SOP No. HW-3a, Revision 1”* (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the

absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JD16644A

The data validation of the metals analytical data in SDG JD16644A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ ICP Interference Check Sample
- √ Data qualifiers
- √ Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Serial dilution analysis
- √ Data package completeness

The two soil samples were analyzed for the five target EPA Method 6010D metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids, were covered by this data validation. Of the sample metals results detected in the two soil samples of SDG JD16644A, no results in the two samples exhibited a concentration above the IGWSSL or SRS, whichever was more stringent.

Laboratory Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. Sample JD16644-1A was diluted for chromium, thallium, and vanadium analysis due to high interfering elements. All other QC requirements for the analytes reviewed for data validation were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130%)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Hence, no sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks and the continuing calibration blanks (CCBs) at the stated reporting limit (RL) or contract required detection limit (CRDL).

No soil sample results warranted qualification for any associated QC blank contamination in SDG JD16644A.

ICP Interference Check Samples (QC Limits: 80-120%)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; $\leq 35\%$ RPD)

The matrix spike (MS) and matrix spike duplicate (MS/MSD) recoveries were within the QC limits of 75 - 125% for the five target analytes in the non-client QC sample JD16703-2 in QC batch MP23962. Hence no sample results were subject to qualification for any matrix spike recovery issues, thereby demonstrating acceptable accuracy.

Duplicate analysis (QC Limit: $\leq 35\%$ RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples from non-client sample JD16703-2. All %RPD values for the 5 target analytes were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 1.1 – 8.9%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated acceptable analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging 90.3% - 99.5% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: $\leq 10\%$ D)

The serial dilution results associated with the soil samples ranged 0 – 6.8%D, values below the QC limit of 10%D criterion for data validation qualification (USEPA, 2017). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

The case narrative did identify that there was one sample (JD16644-1A) with elevated reporting limits for chromium, thallium, and vanadium that were diluted due to the presence of “high interfering element”. Review of the data indicated that the reporting limits were below the respective IGWSSL and SRS limits. No other samples were diluted in the metals analysis, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Metals Results

The soil sample analytical results for the samples of SDG JD16644A were found to be compliant with the analytical methods for the analysis of metals in the two soil samples using SW-846 Method 6010D.

No soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JD16644

The analysis for hexavalent chromium (Cr⁺⁶) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the two (2) soil samples.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Data package completeness | √ Data qualifiers |

Hexavalent chromium was detected in one of the 2 post-excavation soil samples analyzed initially in SDG JD16644. No soil sample Cr+6 results in the samples of SDG JD16644 exceeded the soil cleanup criterion (SCC) of 20 mg/kg, with results being less than 2 mg/kg when detected.

Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery in QC Batch GP30980 was outside control limits, while the insoluble MS recovery and post spike recoveries were within control limits. The RPD value for the duplicate analysis in QC Batch GP30980 was outside control limits, but was acceptable due to low duplicate and sample concentrations. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% CCV Recovery)

The initial calibration demonstrated an acceptable correlation coefficient with a value of 0.99992 for the soil sample analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standard recoveries were all 92.6% for the QC batch associated with the 2 soil samples, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg) or the continuing calibration blanks. Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP30980 associated with 2 soil samples, as presented below in Table 2. Thus, the hexavalent chromium

results in soil samples associated with QC Batch GP30980 required qualification based on the result of the soluble MS recovery due to a potential low bias in the ability to recover Cr⁺⁶ in the associated sample matrices. The insoluble MS recovery was within QC limits and the post spike also recovered within QC limits.

Table 2. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JD16644

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP30980 ¥	JD16644-1	Cr ⁺⁶ , soluble	35.1 %	NJ-/NR	Low
GP30980 ¥	JD16644-1	Cr ⁺⁶ , insoluble	94.6 %	----	----
GP30980 ¥	JD16644-1	Cr ⁺⁶ , post-digestion spike	93.1 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low. NR – The matrix spike sample recovery in the associated QC sample is below the 50% QC limit recommending rejection of the [non-detect] result. ¥ – The samples associated with QC Batch GP30980 consist of JD16567-1 through -4 (inclusive).					

The matrix spike (MS) recovery of 94.6% for the insoluble spike was acceptable, while the soluble MS recovery (35.1%) for the spiked sample JD16644-1 from soil sampling location FNC-4A was below the lower QC limit of 75%, as well as below the 50% criterion where NJDEP DV guidelines (NJDEP, 2009) recommend rejection of associated Cr+6 results. However, professional judgement was applied in not rejecting the results in the associated samples for various reasons discussed below in the Summary section at the end of the Cr+6 review.

Thus, the Cr⁺⁶ results for the 2 samples of this QC batch in SDG JD16644 were qualified following the DV review and flagged with “NJ-” due to a potential low bias in the ability to recover hexavalent chromium from the soil sample matrix. The qualified Cr+6 results are presented below in Table 3.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JD16644-1. The difference between the duplicate soil sample aliquots for Cr+6 in soil this PPG sample FNC-4A was listed as 200.0%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010). However, the difference between the sample (0.45 mg/kg) and its duplicate (0.0 mg/kg) was a value less than two times the reporting limit of < 0.48 mg/kg, the QC limit when either concentration is less than five times the reporting limit. The %RPD values for redox potential (9.2%RPD) and pH (1.2%RPD) displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was within the QC limit of “< 2 × 0.48” mg/kg for soil samples, the associated sample results in the initial analysis are not subject to qualification and represent acceptable analytical precision.

Laboratory Control Sample Analysis (QC Limits: 80-120%)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 85.0% and 93.0% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr⁺⁶ concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr⁺⁶ results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where: A = concentration from calibration curve (mg/L)
- B = Final digested volume (L)
- C = Wet weight of sample (Kg)
- D = % Solids/100
- E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample FNC-4B (JD16644-2) was listed as 1.5 mg/kg on the reporting form and 0.031 mg/L on the quantitation report in the raw data for an undiluted sample. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.031 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00247 \text{ Kg} \times 83.2/100} = \frac{0.0031}{0.0020550} = 1.5085 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 1.5 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium concentration of 1.5 mg/kg for Sample FNC-4B was correctly reported. This was the one detected Cr+6 concentration of the two soil samples analyzed in this SDG, a value considerably below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard mV solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Both of the two soil samples were observed to fall below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The total chromium concentrations of the samples falling within the “reducing” soil conditions were all less than 70 mg/kg with Cr+6 results less than 2 mg/kg. This is not inconsistent with a study of chromium contaminated soils in New

Jersey demonstrated that the general ratio of chromium to Cr+6 was typically a ratio of 20:1 (Paustenbach, et al, 1991). Review of the vast amount of previous PPG data suggests that soil samples containing less than 500 mg/kg total chromium are associated with Cr+6 concentrations less than 20 mg/kg.

Thus, it appears that the Eh-pH phase diagram presents Redox conditions consistent with expectations of the relationship between total chromium and Cr+6, where total chromium concentrations below 70 mg/kg in reducing soil conditions are unlikely to oxidize to Cr+6 concentrations that could approach the SCC of 20 mg/kg.

Summary for Hexavalent Chromium Analysis – SDG JD16644

The matrix spike (MS) recovery of 94.6% for the insoluble spike was acceptable, while the soluble MS recovery (35.1%) for the spiked sample JD16644-1 from soil sampling location FNC-4A was below the lower QC limit of 75%, as well as below the 50% criterion where NJDEP DV guidelines (NJDEP, 2009) recommend rejection of associated Cr+6 results. However, professional judgement was applied in not rejecting the results in the associated samples for various reasons.

Rarely do DV guidelines recommend rejection of detected concentration results, but rather recommend qualification as estimated values. In DV guidance for inorganic analytes (USEPA, 2017), rejection of results is recommended when MS recoveries fall below 30% for non-detect results, but recommend qualification as estimated values (flag with “J”) for detected concentrations. Secondly, the insoluble recovery was well within QC limits and may be a better representation of the ability to recover Cr+6 from a soil matrix, rather than the soluble MS recovery result. Thirdly, because the total chromium result in the associated sample exhibiting a non-detect Cr+6 result (JD16567-2) was 65.7 mg/kg, it is unlikely that Cr+6 results would approach the SCC of 20 mg/Kg, since all samples of this SDG were represented by “reducing” soil conditions which are unfavorable for oxidation of chromium to Cr+6. Additionally, the post-digestion spike was also acceptable (94.8%).

Although the soluble MS recovery in the reanalysis was below the 50% criterion where results are subject to rejection according to DV guideline recommendations, based on professional judgement, the results were merely qualified as estimated values and flagged with “NJ-” rather than rejected. The decision not to reject the soil sample Cr+6 results was based on the following: the reducing nature of the soil conditions for the two samples, the very low total chromium concentrations in the samples, and the favorable insoluble MS recovery, a result that is more likely representative of the analytical accuracy for a soil matrix.

The qualified soil sample results from the Cr+6 analysis in SDG JD16644 are presented below in Table 3. The analytical Cr⁺⁶ results for samples JD16644-1 and JD16644-2 are qualified as estimated values (NJ-) due to a potential low bias suggested by the low soluble MS recovery results, although the insoluble MS analysis exhibited an excellent recovery. The Cr⁺⁶ concentrations determined during the analysis of samples in SDG JD16644 are of similar low magnitude and well below the SCC of 20 mg/kg.

Table 3. Qualified Sample Cr+6 Results in SDG JD16644

Client ID	Laboratory Sample ID	Analyte	Result (mg/kg)	DV Qualifier
FNC-4A	JD16644-1	Cr+6	< 0.48	NJ-
FNC-4B	JD16644-2	Cr+6	1.5	NJ-

Client ID	Laboratory Sample ID	Analyte	Result (mg/kg)	DV Qualifier
< –The analyte was analyzed for, but was not detected above the stated reporting limit. NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.				

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

Paustenbach, D.J., Rinehart, W.E., and P.J. Sheehan, 1991, "The Health Hazards Posed by Chromium-Contaminated Soils in Residential and Industrial Areas: Conclusions of an Expert Panel" in ***Regulatory Toxicology and Pharmacology***, 13, pp 195-222 (1991).

US EPA, CLP, 2017, ***"National Functional Guidelines for Inorganic Superfund Methods Data Review"***, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017.

US EPA, 2016, ***ICP-AES Data Validation, SOP HW-3a, Revision 1***, December 2016.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD16644/JD16644A

1. Were the appropriate sample preservation requirements met?..... Yes No

2. Were appropriate sample holding times (for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

Sample JD16644-1A was diluted for chromium, thallium, and vanadium analysis, due to the presence of high interfering elements.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.
- Not applicable**
10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
20. Provide a brief explanation, if applicable.

Refer to DV report table 2 for QC details. Qualified sample results are presented in Table 3 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDG JD16711/JD16711A
Sample Dates: November 23, 2020
Analyses: Metals Analysis, EPA Method 6010D
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: January 18, 2021

This data validation (DV) report presents the data review and result qualifications for two (2) soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey, on November 23, 2020, for sample delivery group (SDG) JD16711 and JD16711A. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD16711A and JD16711 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium, respectively, in the 2 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JD16711-1A and JD16711-2A

No other sample results in SDG JD16711A and JD16711 required qualification, based on the acceptable remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Impact to Groundwater Soil Screening Level (IGWSSL) and Residential Soil Remediation Standard (SRS) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) in the two samples were both non-detect concentrations and, therefore, were below the Soil Cleanup Criterion (SCC). A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 3 of this DV report.

Sample Receipt

The two (2) soil samples collected November 23, 2020 were received intact and appropriately

preserved the same day, November 23, 2020, at the SGS laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 1.8 degrees Celsius (°C). The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD16711A and JD16711

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
FNC-10A	JD16711 -1A	11/23/2020	Soil	Metals
FNC-10B	JD16711-2A	11/23/2020	Soil	Metals
FNC-10A	JD16711 -1	11/23/2020	Soil	Cr+6
FNC-10B	JD16711-2	11/23/2020	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010D at SGS Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JD16711A, while the data package for the hexavalent chromium analyses is numbered JD16711.

Data Review

Data, as presented in the analytical data packages SDG JD16711A and JD16711 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on US EPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Superfund Methods Data Review*”, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA “*ICP-AES Data Validation, SOP No. HW-3a, Revision 1*” (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data

package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JD16711A

The data validation of the metals analytical data in SDG JD16711A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The two soil samples were analyzed for five target EPA Method 6010D metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids, which were covered by this data validation. Of the sample metals results detected in the 2 samples of SDG JD16711A, no sample metals results exhibited a concentration above the IGWSSL or SRS, whichever was more stringent.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC batch MP23964 indicating possible matrix interference and/or sample nonhomogeneity for the 2 soil samples analyzed in this SDG. The case narrative stated that the RPD serial dilution results for antimony and thallium were outside control limits in QC Batch MP23964, however, the percent difference (%D) results were acceptable due to a low initial sample concentrations (< 50 times instrument detection limit [IDL]). All other QC requirements were met for the target analytes of this SDG, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130% Recovery)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Consequently, no soil sample results were qualified for any calibration issues.

Quality Control Blanks (QC Limit: < Contract Required Detection Limit [CRDL] or < Reporting Limit [RL])

There were no target metals concentrations detected in the procedure blank or continuing calibration blanks (CCBs) at the stated reporting limits. Hence, no soil sample results warranted qualification for any associated QC blank contamination in SDG JD16711A.

ICP Interference Check Samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

(QC Limits: 75-125% Recovery; ≤ 35%Relative Percent Difference [RPD])

The matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were below the QC limits of 75 - 125% in the non-client QC sample JD16648-28 in QC Batch MP23964, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (US EPA, 2017; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP23964 Ω	JD16648-28	Antimony	64.5 %	66.5 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias. Ω – The samples associated with QC Batch MP23964 consist of JD16711-1A and JD16711-2A.						

The antimony results in the two affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate sample aliquots. All %RPD values were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging from 0.0 – 3.6%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated very good analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging from 91.3% – 96.4% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %Difference [%D])

The case narrative stated that the RPD serial dilution results for antimony and thallium were outside control limits in QC Batch MP23964, however, the percent difference (%D) results were acceptable due to a low initial sample concentration (< 50 times IDL) of these two analytes. The serial dilution results for the remaining three analytes associated with the soil samples ranged from 6.7 – 7.8%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2017). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution for the 5 target analytes of this SDG, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JD16711A were found to be compliant with the analytical methods for the analysis of metals in the 2 soil samples using SW-846 Method 6010D.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in the QC batch associated with the 2 soil samples of this SDG, as detailed below in Table 3. The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias in the ability to recover antimony in the affected samples, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JD16711A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
FNC-10A	JD16711-1A	Antimony	< 2.2	NJ-
FNC-10B	JD16711-2A	Antimony	< 2.3	NJ-

Key:
 < –The analyte was analyzed for, but was not detected above the stated reporting limit.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JD16711

The analysis for hexavalent chromium (Cr+6) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the 2 soil samples.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- √ Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Data qualifiers

Hexavalent chromium was not detected in either of the two soil samples analyzed in SDG JD16711, thus, no soil sample Cr+6 results of this SDG exceeded the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Laboratory Case Narrative

The case narrative stated that the RPD for the duplicate analysis for hexavalent chromium was outside control limits, but the RPD was acceptable due to low duplicate and sample concentrations. The case narrative also stated that the matrix spike recoveries are outside control limits. However, the recoveries of 76.9% and 94.3% observed on the Matrix Spike Results Summary form are actually within the QC limits of 75% - 125%. Hence, the QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification [CCV] Standard Recovery)

The initial calibration demonstrated an acceptable correlation coefficient ('r') with a value of 0.99992 for the soil samples analysis, a value greater than the calibration requirement for linearity of 0.995. Calibration check standards recovered with values ranging from 90.4% to 90.7% for the QC batch associated with the 2 soil samples, thereby meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or <RL)

Hexavalent chromium was not detected in the method blank (< 0.40 mg/kg) or the continuing calibration blanks. Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

Despite the statement in the case narrative that "matrix spike recovery(s) are outside control limits", the matrix spike (MS) recoveries for Cr+6 were all within the QC limits of 75 - 125% for PPG sample FNC-10B in QC Batch GP31002, as depicted in Table 4, such that no sample Cr+6 results required qualification for matrix spike recovery results indicating acceptable analytical accuracy.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JD16711

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP31002 ¥	JD16711-2	Cr ⁺⁶ , soluble	76.9 %	----	----
GP31002 ¥	JD16711-2	Cr ⁺⁶ , insoluble	94.3 %	----	----
GP31002 ¥	JD16711-2	Cr ⁺⁶ , post-digestion spike	106 %	----	----
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium ¥ – The samples associated with QC Batch GP31002 consist of JD16711-1 and JD16711-2.					

The Cr+6 results in the two associated soil samples are not subject to qualification and the MS recovery results demonstrate acceptable accuracy.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JC16711-2. The difference between the duplicate soil sample aliquots for Cr+6 in soil this sample (FNC-10B) was listed as 200.0%RPD, a value above the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010). However, the difference between the sample (0.0 mg/kg) and its duplicate (0.39 mg/kg) was a value less than two times the reporting limit of < 0.45 mg/kg. The %RPD values for redox potential (2.5%RPD) and pH (3.2%RPD) displayed acceptable analytical precision results. Because the %RPD value for Cr+6 was within the QC limit of “< 2 × 0.45” mg/kg for soil samples, the associated sample results are not subject to qualification and represent acceptable analytical precision.

Laboratory Control Sample Analysis (QC Limits: 80-120% Recovery)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 92.5% and 95.0% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr+6 concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr+6 results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

- Where:
- A = concentration from calibration curve (mg/L)
 - B = Final digested volume (L)
 - C = Wet weight of sample (kg)
 - D = % Solids/100
 - E = Dilution (if necessary)

The non-detect hexavalent chromium result for Sample FNC-10B (JD16711-2) was listed as < 0.45 mg/kg on the reporting form and 0.0037 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0037 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00251 \text{ Kg} \times 88.1/100} = \frac{0.00037}{0.0022113} = 0.1673 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 0.17 \text{ mg/kg}$$

The reporting limit is determined by: $\frac{\text{mg/kg}}{\text{TS}}$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 0.40 \text{ mg/kg} / 0.881 = < 0.454 \text{ mg/kg} = < 0.45 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the hexavalent chromium result of 0.17 mg/kg for FNC-10B was less than the reporting limit of 0.45 mg/kg and was correctly reported as a non-detect result. Hence, the result is a value below the SCC of 20 mg/kg.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard millivolt (mV) solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

The two soil samples (JD16711-1 and JD16711-2) were on or marginally above the Eh-pH phase line depicting oxidizing conditions. However, since the samples contained non-detect Cr+6 concentrations and exhibited corresponding total chromium results of only 18.5 and 11.0 mg/kg, the Cr+6 concentrations in these two samples are not expected to approach the SCC of 20 mg/kg, since the total chromium concentrations are both below 20 mg/Kg.

Summary for Hexavalent Chromium Analysis – SDG JD16711

Since the MS recoveries were within QC limits, as were all other QC results associated with the hexavalent chromium analysis, including the duplicate sample analysis, no Cr+6 results were qualified following the DV review and are usable as reported.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on US EPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, CLP, 2017, ***“National Functional Guidelines for Inorganic Superfund Methods Data Review”***, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017.

US EPA, 2016, ***ICP-AES Data Validation, SOP HW-3a, Revision 1***, December 2016.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD16711/JD16711A

1. Were the appropriate sample preservation requirements met?..... Yes No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? Yes No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes No
Indicate the identity of the samples and why.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes No
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? Yes No
If "Yes", include the number of samples and laboratory sample ID numbers.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... Yes No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... Yes No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No

9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**

If “yes”, please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds
of concern at the site met? Yes **No**

12. Were the QC Summary Forms reviewed?..... **Yes** No

13. Internal Standards acceptable..... **Yes** No

14. MS/MSD acceptable..... Yes **No**

15. Calibration summaries acceptable..... **Yes** No

16. Serial dilutions acceptable..... **Yes** No

17. Inorganic duplicates acceptable..... **Yes** No

18. LCS recovery acceptable..... **Yes** No

19. Other QC acceptable?..... **Yes** No
Provide a brief explanation, if applicable.

Refer to DV report tables 2 and 4 for QC details. Qualified sample results are presented in Table 3 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JD16870/JD16870A
Sample Dates: November 25, 2020
Analyses: Metals Analysis, EPA Method 6010D
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: January 20, 2021

This data validation (DV) report presents the data review and result qualifications for six (6) soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey on November 25, 2020 for sample delivery group (SDG) JD16870, as well as JD16870A. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD16870A and JD16870 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 6 collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JD16870-1A through JD16870-6A (inclusive);
- Hexavalent chromium ("NR") in Samples JD16870-1 and JD16870-2 (inclusive);
- Hexavalent chromium ("NJ-") in Samples JD16870-3 through JD16870-6 (inclusive)
- Hexavalent chromium ("NJ-") in reanalysis samples JD16870-1R through JD16870-6R (inclusive)

No other sample results in SDG JD16870A and JD16870 required qualification, based on the acceptable remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Residential Soil Remediation Standard (SRS) and Impact to Groundwater Soil Screening Level (IGWSSL) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) of 20 mg/kg in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 3 and Table 6 of this DV report.

Sample Receipt

The six (6) soil samples collected November 25, 2020 were received intact and appropriately preserved November 25, 2020 at the SGS laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 1.7°C. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD16870A and JD16870

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
FNC-6A	JD16870-1A	11/25/2020	Soil	Metals
FNC-6B	JD16870-2A	11/25/2020	Soil	Metals
FNC-5A	JD16870-3A	11/25/2020	Soil	Metals
FNC-5B	JD16870-4A	11/25/2020	Soil	Metals
FNC-7A	JD16870-5A	11/25/2020	Soil	Metals
FNC-7B	JD16870-6A	11/25/2020	Soil	Metals
FNC-6A	JD16870-1	11/25/2020	Soil	Cr+6
FNC-6B	JD16870-2	11/25/2020	Soil	Cr+6
FNC-5A	JD16870-3	11/25/2020	Soil	Cr+6
FNC-5B	JD16870-4	11/25/2020	Soil	Cr+6
FNC-7A	JD16870-5	11/25/2020	Soil	Cr+6
FNC-7B	JD16870-6	11/25/2020	Soil	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010D at SGS Laboratories in Dayton, NJ, as well as percent total solids.				
Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JD16870A, while the data package for the hexavalent chromium analyses is numbered JD16870.

Data Review

Data, as presented in the analytical data packages SDG JD16870A and JD16870 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) *“National Functional Guidelines for Inorganic Superfund Methods Data Review”*, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA *“ICP-AES Data Validation, SOP No. HW-3a, Revision 1”* (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and QC forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JD16870A

The data validation of the metals analytical data in SDG JD16870A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The six soil samples were analyzed for the five target EPA Method 6010D metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids, were covered by this data validation. Of the sample metals results detected in the 6 samples of SDG JD16870, no results exhibited a concentration above the IGWSSL or SRS, whichever was more stringent.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC Batch MP24020 indicating possible matrix interference and/or sample nonhomogeneity in the soil samples analyzed in this SDG. All other QC requirements for the analytes reviewed for data validation were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130%)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Hence, no sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks or the continuing calibration blanks (CCBs) at the stated reporting limit (RL) or contract required detection limit (CRDL). No soil sample results warranted qualification for any associated QC blank contamination in SDG JD16870A.

ICP Interference Check Samples (QC Limits: 80-120%)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis (QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were below the QC limits of 75 - 125% for the PPG QC batch sample JD16858-1, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (USEPA, 2017; NJDEP, 2002). The remaining matrix spike results fell within QC limits.

Table 2. Matrix Spike Recovery Results outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP24020 Ω	JD16858-1	Antimony	74.1 %	72.4 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias. Ω – The samples associated with QC Batch MP24020 consist of JD16870-1A through -6A (inclusive).						

The antimony results in the six affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples from JD16858-1. All %RPD values for the 5 target analytes were below the laboratory QC limit of 20%RPD, with values ranging 2.2 – 4.3%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated excellent analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging 92.3% - 98.9% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The serial dilution results associated with the soil samples ranged 0 – 1.3%D, values below the QC limit of 10%D criterion for data validation qualification (USEPA, 2017). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples were diluted such that the reporting limits were all below the respective IGWSSL or SRS limit values.

Hence, all reporting limits were below the respective project IGWSSL and SRS limit values.

Summary of Qualified Metals Results in JD16870A

The soil sample analytical results for the samples of SDG JD16870A were found to be compliant with the analytical methods for the analysis of metals in the 6 soil samples using SW-846 Method 6010D.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in the QC batch associated with the 6 soil samples of this SDG, as detailed below in Table 3. The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias in the ability to recover antimony in the affected samples, as listed below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JD16870A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
FNC-6A	JD16870-1A	Antimony	< 2.4	NJ-
FNC-6B	JD16870-2A	Antimony	< 2.4	NJ-
FNC-5A	JD16870-3A	Antimony	< 2.4	NJ-
FNC-5B	JD16870-4A	Antimony	< 2.3	NJ-
FNC-7A	JD16870-5A	Antimony	< 2.4	NJ-
FNC-7B	JD16870-6A	Antimony	< 2.3	NJ-

Key:

< –The analyte was analyzed for, but was not detected above the stated reporting limit.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JD16870

The analysis for hexavalent chromium (Cr⁺⁶) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the 6 soil samples. The soil samples were re-analyzed in an additional QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

√ Holding times	Matrix spike recoveries
√ Blank Analysis	√ Duplicate analysis
√ Calibration standards	√ Laboratory control samples
√ Calibration verification	√ Quantitation checks
√ Data package completeness	√ Data qualifiers

Hexavalent chromium was detected in four of the six soil samples initially analyzed in SDG JD16870 with Cr+6 concentrations less than or equal to 2.8 mg/kg, with Cr+6 detected in five of the six samples in the re-analysis with results less than 6 mg/kg in the reanalysis effort. Hence, all sample Cr+6 results are less than the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble and insoluble matrix spike recoveries in QC Batch GP31045 were outside control limits, thereby suggesting that the recoveries indicate possible matrix interference, but the post spike recovery was good. The soluble matrix spike recovery in reanalysis QC Batch GP31359 was also outside control limits, but the post spike recovery was acceptable. The RPDs for Cr+6 in the duplicate analysis of the initial and reanalysis were outside control limits, but the RPDs were acceptable due to low duplicate and sample concentrations. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% CCV Recovery)

The initial calibrations demonstrated acceptable correlation coefficients with a value of 0.99995 for the soil sample analysis a value greater than the calibration requirement for linearity of 0.995, as was the 0.99998 correlation coefficient in the reanalysis. The calibration check standard recoveries were all 90.4% for the three CCVs in the QC batch associated with the 6 soil samples, all meeting the continuing calibration QC requirement of 90-110%. The calibration check standard recoveries were 92.46% for the QC batch associated with the reanalysis of the 6 soil samples.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg), the continuing calibration blanks. Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble and insoluble matrix spike recoveries were below the QC limits of 75-125% for QC Batch GP31045 associated with 6 soil samples, with recoveries of 0.9% and 70.7%, respectively, as presented below in Table 4. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP31045 are subject to rejection based on the results of the soluble MS recovery below 50% and the and insoluble MS recovery below 75%, as recommended in the DV guidelines

for Cr+6 analysis (NJDEP, 2009) and the perceived inability to recover Cr+6 in the associated sample matrices. However, the post spike recovered within the QC limits of 85-115%.

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JD16870

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP31045 ¥	JD16870-1	Cr ⁺⁶ , soluble	0.9 %	NR	Low
GP31045 ¥	JD16870-1	Cr ⁺⁶ , insoluble	70.7 %	NJ-	Low
GP31045 ¥	JD16870-1	Cr ⁺⁶ , post-digestion spike	87.6 %	---	---
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium N – The matrix spike sample recovery in the associated QC sample is outside QC limits R – The result is rejected because the MS recovery in the associated QC sample is below 50%; J- – The result is qualified as an estimated value with a potential low bias; ¥ – The samples associated with QC Batch GP31045 consist of JD16870-1 through -6 (inclusive).					

The non-detect Cr⁺⁶ results associated with the soluble MS recovery below 50% are rejected and flagged with “NR”, as tabulated below in Table 6, together with the qualified results for the four detected Cr+6 results in the initial analysis and the six qualified results (NJ-) in the six samples from the re-analysis of this QC batch.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JD16870-1. The difference between the duplicate soil sample aliquots for Cr+6 in soil this sample (FNC-6A) was listed as 200.0%RPD, a value above the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010). However, the result was acceptable because the sample and its duplicate aliquot were less than five times the CRDL, the QC limit becomes ± 2 x CRDL or two times the respective reporting limit. Thus, the difference between the detected Cr+6 results in JD16870-1 (0.46 mg/kg) and the non-detect result in the duplicate (0.0 mg/kg) was less than the threshold criterion of 0.92 mg/kg. The %RPD values for redox potential (3.5%RPD) and pH (3.5%RPD) displayed acceptable analytical precision results. Hence, the sample Cr+6 results in the samples of this SDG are not subject to qualification, as the duplicate analysis met the alternate QC requirement for low concentration samples and represent acceptable analytical precision.

Laboratory Control Sample Analysis (QC Limits: 80-120%)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries ranging from 88.0% to 101.3% associated with the soil samples, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr⁺⁶ concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr⁺⁶ results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)
 C = Wet weight of sample (Kg)
 D = % Solids/100
 E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample FNC-7A (JD16870-5) was listed as 2.8 mg/kg on the reporting form and 0.0580 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0580 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00252 \text{ Kg} \times 82.6/100} = \frac{0.00580}{0.002082} = 2.7885 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 2.8 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the detected hexavalent chromium result of 2.8 mg/kg for Sample FNC-7A was correctly reported. This was one of the four detected Cr+6 concentrations for the initial analysis of the 6 soil samples of this SDG.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard mV solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Each of the 6 soil samples were observed to fall considerably below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The total chromium concentrations of the samples falling below the Eh-pH phase line representing “reducing” soil conditions were very low, ranging from 13.0 mg/kg to 63.3 mg/kg, thereby extremely limiting the possibility of converting total chromium to Cr+6 to levels approaching the SCC of 20 mg/kg.

Summary for Initial Hexavalent Chromium Analysis – SDG JD16870

Since the soluble MS spike recovery of 0.9% was below QC limits in the QC sample of QC Batch GP31045, the soil samples in this QC batch required reanalysis. The 70.7% insoluble MS

recovery was below the respective QC limit of 75-125%, as depicted in Table 4. Therefore, the non-detect Cr⁺⁶ results for 2 of the 6 samples of this QC batch in SDG JD16870 were subject to rejection following the DV review and flagged with “NR” due to a potential inability to recover hexavalent chromium from the soil sample matrix. Consequently, the soil samples of this QC batch were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analyses in SDG JD16870”.

Cr+6 Re-analyses in SDG JD16870

Because the soluble MS recovery was below QC limits in the initial QC batch triggering reanalysis, the resultant data for the reanalysis of the 6 soil samples (JD16870-1R through -6R) are summarized in this section.

The QC requirements were met during the reanalysis of samples JD16870-1R through -6R in QC Batch GP31359, including the calibrations (r = 0.999978, 92.47% CCV Recoveries), QC blanks, duplicate analysis (200.0 %, but < 2 x RL), and blank spike analysis (92.8% – 97.9%). The 0.9% soluble MS recovery in the initial analysis improved considerably to a recovery of 65.1%, as the 70.7% insoluble MS recovery improved to 94.3% and the post spike recovery (96%) also improved from an 88% result in the initial analysis, as the spike recoveries in the reanalysis are detailed below in Table 5.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble MS and insoluble MS recoveries were both considerably improved in the reanalysis, thereby supporting the decision to reject the tow non-detect Cr+6 sample results in the initial analysis. The following matrix spike recoveries were observed during the reanalysis of the affected samples (Table 5).

Table 5. Hexavalent Chromium Re-analysis MS Recovery Results – JD16870

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP31359 Ж	JD16870-1R	Cr ⁺⁶ , soluble	65.1 %	NJ-	Low
GP31359 Ж	JD16870-1R	Cr ⁺⁶ , insoluble	94.3 %	---	---
GP31359 Ж	JD16870-1R	Cr ⁺⁶ , post-digestion spike	96.0 %	----	---
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium Ж – The samples associated with QC Batch GP31359 consist of JD16870-1R through -6R (inclusive).					

Since the soluble MS recovery in QC Batch GP31359 is 65.1%, the non-detect Cr⁺⁶ results for the samples in this QC batch are no longer subject to rejection, but are qualified as estimated values flagged wrih NJ- due to a potential low bias in the ability to recover Cr⁺⁶ in this QC batch. The qualified Cr+6 results of the reanalysis are presented below in Table 6 together with the results of the initial Cr+6 analysis.

Summary for Hexavalent Chromium Analysis – SDGs JD16870

The qualified (rejected) soil sample results from the initial Cr+6 analysis in SDG JD16870 are presented below in Table 6 alongside those qualified results obtained from the reanalysis of samples in this SDG. Although the initial analytical non-detect Cr⁺⁶ results for samples JD16870-1 and JD16870-2 are subject to rejection (“NR”) due to the very low soluble spike recovery of 0.9%, the results of the reanalysis are only subject to qualification, since the soluble MS recovery

improved to 65.1%, a value below the QC limit of 75% but above 50% where Cr+6 results are to be rejected, as recommended by NJDEP DV guidelines (NJDEP, 2009). The Cr⁺⁶ concentrations determined during the re-analysis of samples in SDG JD16870, that were performed 15 days later within the 30-day holding time, are not dissimilar from those of the initial analysis, with some double those of the initial analysis, potentially due to the improved MS recoveries, but others similar to or even less than the initial analysis, all still well below the SCC of 20 mg/kg, likely limited by the very low concentrations of total chromium and the reducing soil conditions.

Table 6. Comparison of Qualified Cr⁺⁶ Results in JD16870 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JD16870 Result (mg/kg)	DV Qualifier	Reanalysis Results (mg/kg)	DV Qualifier
FNC-6A	JD16870-1	Cr+6	< 0.48	NR	< 0.48	NJ-
FNC-6B	JD16870-2	Cr+6	< 0.46	NR	1.1	NJ-
FNC-5A	JD16870-3	Cr+6	2.6	NJ-	4.1	NJ-
FNC-5B	JD16870-4	Cr+6	1.4	NJ-	1.1	NJ-
FNC-7A	JD16870-5	Cr+6	2.8	NJ-	5.9	NJ-
FNC-7B	JD16870-6	Cr+6	0.80	NJ-	0.77	NJ-
<p>< –The analyte was analyzed for, but was not detected above the stated reporting limit.</p> <p>N – The matrix spike sample recovery in the associated QC sample is outside QC limits</p> <p>R – The result is rejected because the MS recovery in the associated QC sample is below 50%;</p> <p>NR – The matrix spike sample recovery in the associated QC sample is below the 50% QC limit recommending rejection of the result.</p> <p>NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.</p>						

Although the non-detect Cr+6 results were rejected in two of the 6 soil samples in the initial analysis in accordance with DV guidance (NJDEP, 2009), the guidance also suggests that the Eh-pH results can be referred to for data usability.

Despite the poor MS recoveries, it is possible that the reducing environment conditions exhibited by the Eh-pH phase diagram contribute to the observed non-detect Cr+6 results and may actually reflect somewhat representative concentration results because of the reducing soil conditions that do not favor oxidation of chromium to Cr+6.

Additionally, the results of the reanalysis were rather similar to the non-detect results of the initial analysis, and seemingly irrespective of the total chromium concentration.

Though a case can be made for not rejecting any Cr+6 results due to the extremely low total chromium concentrations < 22 mg/kg and given the “reducing” soil conditions, it is extremely unlikely that Cr+6 concentrations could approach the SCC of 20 mg/kg, the two non-detect Cr+6 results were rejected as a conservative measure due to the extremely low 0.9% soluble MS recovery in the initial analysis. Additionally, detected inorganic sample results are typically merely qualified, not rejected, despite very low associated MS recoveries (USEPA, 2017; NJDEP, 2002). Furthermore, the total chromium to Cr+6 ratios in for the four samples with detected Cr+6 concentrations in the initial analysis range from 16 to 24, ratios which are consistent with the 20:1 ratio observed in a study of chromium-contaminated soils in New Jersey which demonstrated that the general ratio of chromium to Cr+6 was typically a ratio of 20:1 (Paustenbach, et al, 1991). Review of the vast amount of previous PPG data suggests that soil samples containing less than 500 mg/kg total chromium are associated with Cr+6 concentrations less than 20 mg/kg.

The results of this data package are considered usable within the context of the applied qualifications in conjunction with other site information.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
R	The result is rejected because the MS recovery in the associated QC sample is below 50%;

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

Paustenbach, D.J., Rinehart, W.E., and P.J. Sheehan, 1991, "The Health Hazards Posed by Chromium-Contaminated Soils in Residential and Industrial Areas: Conclusions of an Expert Panel" in ***Regulatory Toxicology and Pharmacology***, 13, pp 195-222 (1991).

US EPA, CLP, 2017, ***"National Functional Guidelines for Inorganic Superfund Methods Data Review"***, OSWER Publication 9355.0-135, EPA-540-R-2017-001, January 2017.

US EPA, 2016, ***ICP-AES Data Validation, SOP HW-3a, Revision 1***, December 2016.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD16870/JD16870A

- 1. Were the appropriate sample preservation requirements met?..... **Yes** No

- 2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? **Yes** No
If "No", provide a brief explanation.

- 3. Were the samples diluted? Yes **No**
Indicate the identity of the samples and why.

- 4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable
standards?..... Yes **No**
If "Yes", list the affected samples.

- 5. Were any applicable standards exceeded for any samples? Yes **No**
If "Yes", include the number of samples and laboratory sample ID numbers.

- 6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for
the site?..... **Yes** No
If "No", provide a brief explanation of action taken.

- 7. Were qualifications noted in the non-conformance summary?..... **Yes** No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

- 8. Were qualified data used?..... **Yes** No

- 9. Were rejections noted in the non-conformance summary?..... Yes **No**

Provide a brief explanation.

10. Were rejected data used?..... Yes No
- If "yes", please indicate reasons rejected data were used:
- For Hex Chrome, data were rejected because spike recovery was <50%.
 - Data were rejected due to missing deliverables.
 - Data were rejected but an applicable standard exceedance exists.
 - Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 - Other reasons not noted directly above. Explain:

Sample non-detect Cr+6 results were rejected in the initial analysis because the soluble MS recovery was below 50%. Sample Cr+6 results in the reanalysis are qualified as estimated values, since the MS recovery was above 50%. It is recommended that the reanalysis Cr+6 results be used for reporting.

11. Were the quality control criteria associated with the compounds of concern at the site met? Yes No
12. Were the QC Summary Forms reviewed?..... Yes No
13. Internal Standards acceptable..... Yes No
14. MS/MSD acceptable..... Yes No
15. Calibration summaries acceptable..... Yes No
16. Serial dilutions acceptable..... Yes No
17. Inorganic duplicates acceptable..... Yes No
18. LCS recovery acceptable..... Yes No
19. Other QC acceptable?..... Yes No
20. Provide a brief explanation, if applicable.

Refer to DV report tables 2, 4, and 5 for QC details. Qualified sample results are presented in Tables 3 and 6 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JD16918/JD16918A
Sample Dates: November 30 and December 1, 2020
Analyses: Metals Analysis, EPA Method 6010D
Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: January 25, 2021

This data validation (DV) report presents the data review and result qualifications for five (5) post-excavation soil samples and one (1) field blank (FB) collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey on November 30 and December 1, 2020 for sample delivery group (SDG) JD16918, as well as JD16918A. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD16918A and JD16918 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 5 collected soil samples and one field blank.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JD16918-1A through JD16918-5A (inclusive);

No other sample results in SDG JD16918A and JD16918 required qualification, based on the acceptable remaining associated QC results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Residential Soil Remediation Standard (SRS) and Impact to Groundwater Soil Screening Level (IGWSSL) limits, whichever was more stringent, while the hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) of 20 mg/kg in the respective SDGs. A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 3 of this DV report.

Sample Receipt

The five (5) post-excavation soil samples and one field blank collected November 30 and December 1, 2020 were received intact and appropriately preserved December 1, 2020 at the SGS laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 3.1°C. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD16918A and JD16918

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
FNC-8A	JD16918-1A	11/30/2020	Soil	Metals
FNC-8B	JD16918-2A	11/30/2020	Soil	Metals
DUP	JD16918-3A	11/30/2020	Soil	Metals
FNC-9A	JD16918-4A	12/1/2020	Soil	Metals
FNC-9B	JD16918-5A	12/1/2020	Soil	Metals
FB01	JD16918-6A	12/1/2020	Aqueous	Metals
FNC-8A	JD16918-1	11/30/2020	Soil	Cr+6
FNC-8B	JD16918-2	11/30/2020	Soil	Cr+6
DUP	JD16918-3	11/30/2020	Soil	Cr+6
FNC-9A	JD16918-4	12/1/2020	Soil	Cr+6
FNC-9B	JD16918-5	12/1/2020	Soil	Cr+6
FB01	JD16918-6	12/1/2020	Aqueous	Cr+6
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010D at SGS Laboratories in Dayton, NJ, as well as percent total solids. Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential.				

The data package presenting the metals data is numbered JD16918A, while the data package for the hexavalent chromium analyses is numbered JD16918.

Data Review

Data, as presented in the analytical data packages SDG JD16918A and JD16918 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Superfund Methods Data Review*”, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA “*ICP-AES Data Validation, SOP No. HW-3a, Revision 1*” (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JD16918A

The data validation of the metals analytical data in SDG JD16918A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The five soil samples and one field blank were analyzed for the five target EPA Method 6010D metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids, were covered by this data validation. Of the sample metals results detected in the 5 samples of SDG JD16918A, no results exhibited a concentration above the IGWSSL or SRS, whichever was more stringent.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC Batch MP24053 indicating possible matrix interference and/or sample nonhomogeneity in the soil samples analyzed in this SDG. The case narrative also stated that the relative percent difference (RPD) results for antimony and thallium in the serial dilution analysis for the soil sample analysis were outside control limits in QC Batch MP24053. The thallium result in the serial dilution analysis for the aqueous fraction was outside control limits in QC Batch MP24054. However, the percent difference (%D) results were all acceptable due to the low initial sample antimony and thallium concentrations (< 50 times the

instrument detection limit [IDL]). All other QC requirements for the analytes reviewed for data validation were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130%)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Hence, no sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks, the continuing calibration blanks (CCBs) or field blank at the stated reporting limit (RL) or contract required detection limit (CRDL). No soil sample results warranted qualification for any associated QC blank contamination in SDG JD16918A.

ICP Interference Check Samples (QC Limits: 80-120%)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis (QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were below the QC limits of 75 - 125% for the PPG QC batch sample JD16918-5A, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (USEPA, 2017; NJDEP, 2002). The remaining matrix spike results fell within QC limits, including those of QC Batch MP24054 for the field blank.

Table 2. Matrix Spike Recovery Results outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP24053 Ω	JD16918-5A	Antimony	61.4 %	65.2 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias. Ω – The samples associated with QC Batch MP24053 consist of JD16918-1A through -5A (inclusive).						

The antimony results in the five affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in the summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples from JD16918-5A. All %RPD values for the 5 target analytes were below the laboratory QC limit of 20%RPD, as well as below the project QC limit of 35%RPD for soil samples, with values ranging 0.0 – 5.9%RPD for soil samples with no results requiring qualification, and 0.5 – 1.1% for QC Batch MP24054 associated with the field blank. The duplicate analyses demonstrated acceptable analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging 89.8% - 91.8% for the soil sample metals analysis, and 88.0 – 92.0% for the aqueous fraction for the field blank analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative identified the serial dilution results being outside QC limits for thallium in QC Batch MP24054 associated with the field blank and for antimony and thallium in QC Batch MP24053 associated with the 5 soil samples. The percent difference values were acceptable due to low observed initial sample concentrations (< 50 times IDL).

The remaining serial dilution results associated with the soil samples ranged from 4.9 to 5.8%D, with 0% results for the aqueous fraction, values below the QC limit of 10%D criterion for data validation qualification (USEPA, 2017). No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Hence, all reporting limits were below the respective project IGWSSL and SRS limit values.

Summary of Qualified Metals Results in JD16918A

The soil sample analytical results for the samples of SDG JD16918A were found to be compliant with the analytical methods for the analysis of metals in the 5 soil samples and one field blank using SW-846 Method 6010D.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in the QC batch associated with the 5 soil samples of this SDG, as detailed below in Table 3. The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias in the ability to recover antimony in the affected samples, as listed below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JD16918A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
FNC-8A	JD16918-1A	Antimony	< 2.3	NJ-
FNC-8B	JD16918-2A	Antimony	< 2.5	NJ-
DUP	JD16918-3A	Antimony	< 2.7	NJ-
FNC-9A	JD16918-4A	Antimony	< 2.7	NJ-
FNC-9B	JD16918-5A	Antimony	< 2.4	NJ-

Key:
< –The analyte was analyzed for, but was not detected above the stated reporting limit.
NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

2.0 Hexavalent Chromium Analysis Data Review – SDG JD16918

The analysis for hexavalent chromium (Cr⁺⁶) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the 5 soil samples and one QC batch for the field blank. The soil samples were re-analyzed in an additional QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- √ Holding times
- √ Blank Analysis
- √ Calibration standards
- √ Calibration verification
- √ Data package completeness
- √ Matrix spike recoveries
- √ Duplicate analysis
- √ Laboratory control samples
- √ Quantitation checks
- √ Data qualifiers

Hexavalent chromium was detected in two of the five soil samples initially analyzed in SDG JD16918, with detected Cr+6 concentrations of 2.3 mg/kg in each. Hence, all sample Cr+6 results are less than the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg.

Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. The soluble and insoluble matrix spike recoveries in QC Batch GP31111 were within control limits, as were the post spike recovery and the matrix spike recoveries associated with the aqueous fraction. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% CCV Recovery)

The initial calibrations demonstrated acceptable correlation coefficients with a value of 0.99997 for the soil sample analysis and 0.99982 for the aqueous fraction, values greater than the calibration requirement for linearity of 0.995. The calibration check standard recoveries were 90.3% for the QC batch associated with the 5 soil samples, and ranged from 96.6% to 97.1% for the aqueous fraction, all meeting the continuing calibration QC requirement of 90-110%.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg), the continuing calibration blanks, or the field blank (< 0.010 mg/L). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The matrix spike (MS) recoveries for Cr+6 were all within the QC limits of 75 - 125% for PPG sample FNC-9B in QC Batch GP31111, as depicted in Table 4, such that no sample Cr+6 results required qualification for matrix spike recovery results indicating acceptable analytical accuracy

Table 4. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JD16918

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GN13668 ω	JD16912-1	Cr ⁺⁶ , soluble	100.0 %	---	---
GP31111 ¥	JD16918-5	Cr ⁺⁶ , soluble	82.8 %	---	---
GP31111 ¥	JD16918-5	Cr ⁺⁶ , insoluble	92.0 %	---	---
GP31111 ¥	JD16918-5	Cr ⁺⁶ , post-digestion spike	102 %	---	---
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium ω – The sample associated with QC Batch GN13668 consists of JD16918-6. ¥ – The samples associated with QC Batch GP31111 consist of JD16918-1 through -5 (inclusive).					

The Cr+6 results in the five associated soil samples are not subject to qualification and the MS recovery results demonstrate acceptable accuracy.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one set of duplicate soil samples from sample JD16918-5. The difference between the duplicate soil sample aliquots for Cr+6 in soil this sample (FNC-9B) was listed as 0.0%RPD, a value below the 20%RPD laboratory QC limit, as well as the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010). The RPD value for the aqueous QC Batch GN13668 associated with the field blank was also 0.0%. The %RPD values for redox potential (10.7%RPD) and pH (0.5%RPD) displayed acceptable analytical precision results for the soil sample analysis, as did the respective results of 0.3% and 0.0% for the aqueous fraction. Because the %RPD value for Cr+6 was within the QC limit for soil samples, the associated sample results are not subject to qualification and represent acceptable analytical precision.

Laboratory Control Sample Analysis (QC Limits: 80-120%)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 85.8% and 88.4% associated with the soil samples, and 98.7% for the aqueous fraction associated with the field blank, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr results were qualified for serial dilution analysis results, as it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Sample Result Verification

Sample Cr⁺⁶ concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr⁺⁶ results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where: A = concentration from calibration curve (mg/L)
 B = Final digested volume (L)
 C = Wet weight of sample (Kg)
 D = % Solids/100
 E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample FNC-9A (JD16918-4) was listed as 2.3 mg/kg on the reporting form and 0.0439 mg/L on the quantitation report in the raw data for an undiluted sample. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0439 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00249 \text{ Kg} \times 76.5/100} = \frac{0.00439}{0.0019049} = 2.30464 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 2.3 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the detected hexavalent chromium result of 2.3 mg/kg for Sample FNC-9A was correctly reported. This was one of the two detected Cr+6 concentrations for the initial analysis of the 5 soil samples of this SDG.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard mV solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Each of the 5 soil samples were observed to fall considerably below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions. The total chromium concentrations of the samples falling below the Eh-pH phase line representing “reducing” soil conditions ranged from 21 mg/kg to 107 mg/kg, making it unlikely that oxidation of the soil samples would occur that would approach the SCC of 20 mg/kg.

The reducing environment conditions exhibited by the Eh-pH phase diagram likely contribute significantly to the observed low or non-detect Cr+6 results and may reflect representative results, due to the reducing soil conditions.

Summary for Initial Hexavalent Chromium Analysis – SDG JD16918

Since the MS recoveries were within QC limits, as were all other QC results associated with the hexavalent chromium analysis, including the duplicate sample analysis, no Cr+6 results were qualified following the DV review and are usable as reported.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, CLP, 2017, ***“National Functional Guidelines for Inorganic Superfund Methods Data Review”***, OSWER Publication 9355.0-135, EPA-540-R-2017-001, January 2017.

US EPA, 2016, ***ICP-AES Data Validation, SOP HW-3a, Revision 1***, December 2016.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD16918/JD16918A

1. Were the appropriate sample preservation requirements met?..... **Yes** No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? **Yes** No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes **No**
Indicate the identity of the samples and why.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes **No**
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? Yes **No**
If "Yes", include the number of samples and laboratory sample ID numbers.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... **Yes** No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... **Yes** No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No
9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.
10. Were rejected data used?..... Yes **No**
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:
11. Were the quality control criteria associated with the compounds of concern at the site met? Yes **No**
12. Were the QC Summary Forms reviewed?..... **Yes** No
13. Internal Standards acceptable..... **Yes** No
14. MS/MSD acceptable..... Yes **No**
15. Calibration summaries acceptable..... **Yes** No
16. Serial dilutions acceptable..... **Yes** No
17. Inorganic duplicates acceptable..... **Yes** No
18. LCS recovery acceptable..... **Yes** No
19. Other QC acceptable?..... **Yes** No
20. Provide a brief explanation, if applicable.

Refer to DV report tables 2, and 4 for QC details. Qualified sample results are presented in Table 3 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JD17978/JD17978A
Sample Dates: December 18, 2020
Analyses: Hexavalent Chromium Analysis, EPA Method 3060A/7196A
Redox Potential, ASTM D1498-76M
pH, EPA Method 9045D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga. Ph.D., REP5554
Report Date: January 27, 2021

This data validation (DV) report presents the data review and result qualifications for seven (7) soil samples and one (1) field blank (FB) collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey on December 18, 2020 for sample delivery group (SDG) JD17978. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD17978 were found to be compliant with the analytical methods employed for the analysis of metals and hexavalent chromium in the 7 collected soil samples and one field blank.

Following the detailed DV review, the following sample results were qualified:

- Hexavalent chromium ("NJ-") in Samples JD17978-1;
- Hexavalent chromium ("NR") in Samples JD17978-3 through JD17978-8 (inclusive);
- Hexavalent chromium ("*NJ-") in Samples JD17978-1R, JD17978-3R through JD17978-8R (inclusive).

No other sample results in SDG JD17978 required qualification, based on the acceptable remaining associated quality control (QC) results and analytical performance. Details are provided in the tables and text below. The reported hexavalent chromium (Cr+6) concentrations were all below the Soil Cleanup Criterion (SCC) of 20 mg/kg. A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 7 of this DV report.

Sample Receipt

The seven (7) soil samples and one field blank collected December 18, 2020 were received intact and appropriately preserved December 18, 2020 at the SGS laboratory in Dayton, NJ with

acceptable sampling cooler temperatures with a maximum corrected temperature of 2.1°C. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD17978

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
GGMB-6-17.5	JD17978-1	12/18/2020	Soil	Cr+6
FB-01	JD17978-2	12/18/2020	Aqueous	Cr+6
GGMB-6-20.0	JD17978-3	12/18/2020	Soil	Cr+6
GGMB-6-25.0	JD17978-4	12/18/2020	Soil	Cr+6
GGMB-7-18.0	JD17978-5	12/18/2020	Soil	Cr+6
DUP	JD17978-6	12/18/2020	Soil	Cr+6
GGMB-7-22.0	JD17978-7	12/18/2020	Soil	Cr+6
GGMB-7-24.0	JD17978-8	12/18/2020	Soil	Cr+6
Cr+6 – Hexavalent chromium analyzed by SW-846 Method 7196A together with pH and redox potential, at SGS Laboratories in Dayton, NJ, as well as percent total solids				

The data package presenting the hexavalent chromium analyses is numbered JD17978.

Data Review

Data, as presented in the analytical data packages SDG JD17978 was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods)*, SOP No. 5.A.16 (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) “*National Functional Guidelines for Inorganic Superfund Methods Data Review*”, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA “*ICP-AES Data Validation, SOP No. HW-3a, Revision 1*” (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data package was complete for the hexavalent chromium analysis, and the Cr+6 and associated QC results were substantiated during the DV review. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Hexavalent Chromium Analysis Data Review – SDG JD17978

The analysis for hexavalent chromium (Cr⁺⁶) was performed using US EPA Method 3060A for sample preparation and Method 7196A for sample analysis. The samples were analyzed in one QC batch for the 7 soil samples and one QC batch for the field blank. The soil samples were re-analyzed in an additional QC batch.

The data validation of the analytical data was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements.

- | | |
|-----------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Quantitation checks |
| √ Field duplicate sample analysis | √ Data package completeness |
| √ Data qualifiers | |

Hexavalent chromium was detected in one of the seven soil samples initially analyzed in SDG JD17978, as well as five of the seven samples in the reanalysis effort. All sample Cr+6 results are less than the hexavalent chromium soil cleanup criterion (SCC) of 20 mg/kg, with Cr+ concentrations all less than 4.5 mg/kg.

Case Narrative

The case narrative indicated that the QC requirements were met for issues such as the holding time and method blanks. However, the soluble matrix spike recovery in QC Batch GP31524 was outside control limits, thereby suggesting possible matrix interference. The soluble matrix spike recovery in reanalysis QC Batch GP31602 was also outside control limits. The relative percent difference (RPD) was outside control limits in the duplicate analysis for both the initial and reanalysis due to possible sample nonhomogeneity. All other QC requirements were met for the associated analyses.

Calibrations (r = 0.995; 90-110% Continuing Calibration Verification [CCV] Standard Recovery)

The initial calibrations demonstrated acceptable correlation coefficients with a value of 0.99993 for the soil sample analysis and 0.99995 for the aqueous fraction, values greater than the calibration requirement for linearity of 0.995, as was the 0.99993 correlation coefficient in the reanalysis. The calibration check standard recoveries ranged from 90.2% to 90.5% for the QC batch associated with the 7 soil samples and ranged from 97.6% to 99.5% for the aqueous fraction, all meeting the continuing calibration QC requirement of 90-110%. The calibration check standard recoveries were 93.8% for the QC batch associated with the reanalysis of the 7 soil samples.

Quality Control Blanks (QC Limit: < CRDL or < RL)

Hexavalent chromium was not detected in any of the method blanks (< 0.40 mg/kg), the continuing calibration blanks, or the field blank (< 0.010 mg/L). Thus, no sample results are affected or qualified for any potential QC blank contamination.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble matrix spike recovery was below the QC limits of 75-125% for QC Batch GP31524 associated with 7 soil samples, with a recovery of 23.8%, as presented below in Table 2. Thus, the hexavalent chromium results in soil samples associated with QC Batch GP31524 are subject to rejection based on the results of the soluble MS recovery below 50%, as recommended in the DV guidelines for Cr+6 analysis (NJDEP, 2009) and the perceived inability to recover Cr⁺⁶ in the associated sample matrices. The insoluble MS and post-digestion spike recovered within QC limits.

Table 2. Hexavalent Chromium Analysis Matrix Spike Recovery Results – JD17978

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GN14246 ω	JD17988-6	Cr ⁺⁶ , soluble	100.0 %	---	---
GP31524 ¥	JD17978-1	Cr ⁺⁶ , soluble	23.8 %	NJ-NR	Low
GP31524 ¥	JD17978-1	Cr ⁺⁶ , insoluble	87.3 %	---	---
GP31524 ¥	JD17978-1	Cr ⁺⁶ , post-digestion spike	86.2 %	---	---

QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery
 MS – Matrix spike
 Cr⁺⁶ – Hexavalent chromium
 N – The matrix spike sample recovery in the associated QC sample is outside QC limits
 J- – The reported result is an estimated value with a potential low bias
 R – The result is rejected because the MS recovery in the associated QC sample is below 50%;
 ω – The sample associated with QC Batch GN14246 consists of JD17978-2;
 ¥ – The samples associated with QC Batch GP31524 consist of JD17978-1 and JD17978-3 through -8 (inclusive).

The non-detect Cr⁺⁶ results associated with soluble MS recovery below 50% are rejected and flagged with “NR”, as tabulated below in Table 7. The detected Cr+6 result in sample JD17978-1 was qualified as an estimated value, as detected inorganic results are typically to be qualified as estimated values when associated with MS recoveries below 30% (USEPA, 2017).

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The difference between the duplicate soil sample aliquot concentrations for Cr+6 in PPG sample JD17978-1 was 73.2%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010), as presented below in Table 3. However, the difference was less than 2 × contract required detection limit (CRDL), the QC limit when one or both sample concentrations are less than 5 × CRDL. Thus, the associated sample Cr+6 results were not subject to qualification.

Table 3. Duplicate Analysis Results outside QC Limits

QC Batch	QC Sample	Analyte	Original Result (mg/Kg)	Duplicate (mg/Kg)	Difference	DV Qualifier
GN14246 ω	DA31214-2	Cr+6	0.0 mg/L	0.0 mg/L	0.0	---
GP31524 ¥	JD17978-1	Cr+6	1.4	0.65	73.2 %RPD; < 2 × CRDL	---

QC Limit is 35%RPD or $< 2 \times \text{CRDL}$;

ω – The sample associated with QC Batch GN14246 consists of JD17978-2;

¥ – The samples associated with QC Batch GP31524 consist of JD17978-1 and JD17978-3 through -8 (inclusive).

Since the duplicate analysis results were both less than five times the reporting limit, the QC limit becomes a value of less than two times the reporting limit for soil samples ($\leq 2 \times \text{CRDL}$). Since the difference between the duplicate analysis results is a value of 0.75, this is less than the 0.92 mg/L criterion such that the Cr+6 results in the initial analysis are not subject to qualification.

Laboratory Control Sample Analysis (QC Limits: 80-120%)

The recoveries in the laboratory control samples (LCSs), also referred to as blank spikes, recovered within the 80-120% QC limits, with blank spike recoveries of 90.8% and 92.0% associated with the soil samples, and 96.0% for the aqueous fraction associated with the field blank, thereby demonstrating acceptable analytical system performance.

Serial Dilution Analysis

No sample Cr⁺⁶ results were qualified for serial dilution analysis results. As it appears that a serial dilution analysis was not performed in the analytical sequence. Serial dilution is not a requirement of the analytical method.

Field Duplicate Analysis (QC Limit $\leq 50\%$ RPD)

The results for the analysis of one set of field duplicate samples are presented in Table 4, below. The difference for the non-detect concentrations observed in the field duplicate samples from sampling location GGMB-7-18.0 differed by less than two times the CRDL (reporting level), the QC limit when sample concentrations are less than five times the reporting limit, since the field duplicate sample results were both non-detect concentrations.

Table 4. Comparison of Field Duplicate Soil Sample Results.

Analyte	GGMB-7-18.0 (mg/kg)	DUP (mg/kg)	% RPD	DV Flag
Hex.Chromium	< 0.56	< 0.67	< 2 × CRDL	-

< – The analyte was not detected at the stated reporting limit.
CRDL – The value representing the US EPA CLP contract required detection limit, often represented by the reporting limit;
< 2 × CRDL – The difference between field duplicate results was less than two times the CRDL and meets QC requirements.

The field duplicate results from sampling location GGMB-7-18.0 are not subject to qualification because the difference between the results (0%RPD; $< 2 \times \text{CRDL}$) met the data quality objective and QC limits for sampling, thereby indicating acceptable sampling representativeness and precision for the Cr+6 analysis.

Sample Result Verification

Sample Cr⁺⁶ concentrations reported on the Form 1 (Report of Analysis) sheets for the samples were verified from the raw quantitation reports in the raw data and adjusted for percent solids during the data validation review activity. The following equation was used to verify reported Cr⁺⁶ results:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

Where:

- A = concentration from calibration curve (mg/L)
- B = Final digested volume (L)
- C = Wet weight of sample (Kg)
- D = % Solids/100
- E = Dilution (if necessary)

The detected hexavalent chromium concentration for Sample GGMB-6-17.5 (JD17978-1) was reported as 1.4 mg/kg on the reporting form and 0.0309 mg/L on the quantitation report in the raw data. A calculation check provides the following result:

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{A \times B \times E}{C \times D}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = \frac{0.0309 \text{ mg/L} \times 0.1 \text{ L} \times 1}{0.00255 \text{ kg} \times 85.7/100} = \frac{0.00309}{0.0021854} = 1.41396 \text{ mg/kg}$$

$$\text{Cr}^{+6} \text{ (mg/kg)} = 1.4 \text{ mg/kg}$$

After rounding to two significant figures, this verifies that the detected hexavalent chromium result of 1.4 mg/kg for Sample GGMB-6-17.5 was correctly reported. This was the only detected Cr+6 concentration for the initial analysis of the 7 soil samples of this SDG.

pH/Eh (ORP)

The calibrations for pH analysis were acceptable and the QC requirements were met for duplicate analysis. Standard mV solution checks for Eh analysis were acceptable and within the QC ranges, as were the duplicate sample analyses. The reported pH and Eh results were verified and found to be represented correctly on the Eh/pH phase diagrams. No disparities relative to the reported values and characteristics were observed. All results met the QC limits, such that no pH or redox potential (ORP) results are subject to qualification.

Each of the 7 soil samples were observed to fall considerably below or just below the Eh-pH phase diagram line, thereby suggesting that the samples experience conditions of a “reducing” soil environment. The Cr+6 sample results in a reducing soil are not expected to increase in value because oxidation to Cr+6 is not favorable under the reducing soil conditions.

Summary for Initial Hexavalent Chromium Analysis – SDG JD17978

Since the soluble MS spike recovery of 23.8% was below QC limits in the QC sample of QC Batch GP31524, the soil samples in this QC batch required reanalysis. The 87.3% insoluble MS and 86.2% post-spike recoveries were each within the respective QC limits, as depicted in Table 2. Therefore, the non-detect Cr⁺⁶ results for the 6 samples of this QC batch in SDG JD17978 were rejected following the DV review and flagged with “NR” due to a potential inability to recover hexavalent chromium from the sample matrix. The detected Cr+6 result in JD17978-1 was qualified as an estimated concentration (flagged with NJ-), since DV guidelines recommend qualification of detected results associated with MS recoveries below 30% (USEPA, 2017). Consequently, the soil samples of this QC batch were reanalyzed and the resultant data review is presented in the section below labeled “Cr+6 Re-analyses in SDG JD17978”.

Cr+6 Re-analyses in SDG JD17978

Because the soluble MS recovery was below QC limits in the initial QC batch triggering reanalysis, the resultant data for the reanalysis of the 7 soil samples (JD17978-1R, and JD17978-3R through -8R) are summarized in this section.

The QC requirements were met during the reanalysis of samples JD17978-1R and JD17978-3R through -8R in QC Batch GP31602, including the calibrations (r = 0.99983, 93.8% CCV Recoveries), QC blanks, field duplicate sample results (0%RPD), and blank spike analysis (92.3% – 95.1%). The 55.3% soluble MS recovery in the reanalysis was considerably improved from the initial 23.8% soluble MS recovery in the initial analysis. The insoluble MS and post-spike recoveries were both acceptable and similar to those in the initial analysis, as detailed below in Table 5.

Matrix Spike Analysis (QC Limits: 75-125% Recovery)

The soluble MS recovery of 55.3% was still below the QC range of 75% - 125% in the reanalysis, but above 50%, such that the Cr+6 results in the associated soil samples are all subject to qualification, rather than rejection (NJDEP, 2009). The insoluble MS and post-spike recoveries were acceptable and within the respective QC limits.

The following matrix spike recoveries were observed during the reanalysis of the affected samples (Table 5).

Table 5. Hexavalent Chromium Re-analysis MS Recovery Results – JD17978

QC Batch	QC Sample	Analyte	MS Recovery	DV Qualifier	Potential Bias
GP31602 X	JD17978-1R	Cr ⁺⁶ , soluble	55.3 %	NJ-	Low
GP31602 X	JD17978-1R	Cr ⁺⁶ , insoluble	88.6 %	---	---
GP31602 X	JD17978-1R	Cr ⁺⁶ , post-digestion spike	98 %	----	---
QC Limits are 75-125% for MS recovery; 85-115% for post spike recovery MS – Matrix spike Cr ⁺⁶ – Hexavalent chromium X – The samples associated with QC Batch GP31602 consist of JD17978-1R and JD17978-3R through -8R (inclusive).					

Since the soluble MS recovery in QC Batch GP31602 is below 75%, but above 50%, the Cr⁺⁶ results for the samples in this QC batch are subject to qualification and are flagged with “NJ-” for a potential low bias in the ability to recover Cr⁺⁶ in this QC batch. The qualified Cr+6 results of the reanalysis are presented below in Table 7 together with the results of the initial Cr+6 analysis.

Duplicate Analysis (QC Limit: ≤ 35 %RPD)

The difference between the duplicate soil sample aliquot concentrations for Cr+6 in PPG sample JD17978-1R was 79.4%RPD, a value above the 20%RPD laboratory QC limit, as well as above the 35%RPD QC limit for soil samples (USEPA, 2017; AECOM, 2010), as presented below in Table 6. The difference was also greater than 2 × CRDL, the QC limit when one or both sample concentrations are less than 5 × CRDL. The associated sample Cr+6 results were qualified as estimated values and are to be flagged with “*J” because of potential variability in the analytical precision.

Table 6. Duplicate Analysis Results outside QC Limits

QC Batch	QC Sample	Analyte	Original Result (mg/Kg)	Duplicate (mg/Kg)	Difference	DV Qualifier
GP31602 X	JD17978-1R	Cr+6	4.4	1.9	79.4 %RPD	*J
QC Limit is 35%RPD or < 2 × CRDL; * – Duplicate analysis not within control limits; indeterminate bias direction. J – The reported result is an estimated value. X – The samples associated with QC Batch GP31602 consist of JD17978-1R and JD17978-3R through -8R (inclusive).						

Since the duplicate analysis for Cr+6 differed by more than 35%RPD and the difference is also greater than two times the reporting limit ($2 \times 0.46 = 0.92$ mg/L), the seven associated PPG samples with laboratory ID numbers JD17978-1R and JD17978-3R through JD17978-8R (inclusive) are qualified as estimated values and flagged with the DV qualifier combination “*NJ-” in Table 7 due also to the low associated MS recovery in the batch QC sample.

Summary for Hexavalent Chromium Analysis – SDG JD17978

The qualified and rejected soil sample results from the initial Cr+6 analysis in SDG JD17978 are presented below in Table 7 alongside those qualified results obtained from the reanalysis of samples in this SDG.

The non-detect analytical Cr⁺⁶ results for samples JD17978-3 through -8 (inclusive) of the initial analysis are rejected (“NR”) due to the very low soluble MS spike recovery and the potential inability to recover Cr+6, as recommended by NJDEP DV guidelines (NJDEP, 2009). While Cr⁺⁶ was detected in only one sample in the initial analysis, Cr+6 was detected at low concentrations in five samples of the reanalysis, perhaps reflected by the improved soluble MS recovery during the re-analysis of samples in SDG JD17978, that were performed 7 days later within the 30-day holding time. Though Cr+6 was detected in four more samples in the re-analysis, concentrations were still low, being less than equal to 1.2 mg/kg, as depicted in Table 7 below. Hence, the detected Cr+6 concentrations are still well below the SCC of 20 mg/kg.

Table 7. Comparison of Qualified Cr⁺⁶ Results in JD17978 and Re-analysis

Client ID	Laboratory Sample ID	Analyte	JD17978 Result (mg/kg)	DV Qualifier	Reanalysis Results (mg/kg)	DV Qualifier
GGMB-6-17.5	JD17978-1	Cr+6	1.4	NJ-	4.4	*NJ-
GGMB-6-20.0	JD17978-3	Cr+6	0.60	UNR	1.0	*NJ-
GGMB-6-25.0	JD17978-4	Cr+6	0.64	UNR	0.64	U*NJ-
GGMB-7-18.0	JD17978-5	Cr+6	0.56	UNR	1.2	*NJ-
DUP	JD17978-6	Cr+6	0.67	UNR	1.2	*NJ-
GGMB-7-22.0	JD17978-7	Cr+6	0.67	UNR	0.68	*NJ-
GGMB-7-24.0	JD17978-8	Cr+6	0.66	UNR	0.66	U*NJ-
U –The analyte was analyzed for, but was not detected at the stated reporting limit. N – The matrix spike sample recovery in the associated QC sample is outside QC limits R – The result is rejected because the MS recovery in the associated QC sample is below 50%; NR – The matrix spike sample recovery in the associated QC sample is below the 50% QC limit recommending rejection of the result. * – Duplicate analysis not within control limits; indeterminate bias direction. NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is						

Client ID	Laboratory Sample ID	Analyte	JD17978 Result (mg/kg)	DV Qualifier	Reanalysis Results (mg/kg)	DV Qualifier
estimated and may be biased low.						

Despite the rejection of the non-detect Cr+6 results in six samples of the initial analysis in accordance with DV guidance (NJDEP, 2009), the guidance also suggests that the Eh-pH results can be referred to for data usability. Although the samples appear to be in a “reducing” soil environment, the absence of corresponding total chromium data precludes an evaluation of the usability of the rejected non-detect Cr+6 results that might otherwise support the qualification of the non-detected Cr+6 results as estimated values, rather than their rejection, were it known that total chromium concentrations were, for example, below 100 mg/kg.

Although the soluble MS recovery in the initial analysis was very low, it is likely that the reducing environment conditions exhibited by the Eh-pH phase diagram may contribute to the observed non-detect Cr+6 results and may actually reflect somewhat representative results because of the reducing soil conditions that do not favor oxidation of chromium to Cr+6. Though the Cr+6 concentrations increased in five of the seven samples in the re-analysis, with two results remaining not detected, the Cr+6 concentrations were significantly below the SCC of 20 mg/kg and are not expected to approach the SCC due to the presence of the samples in a reducing soil environment where the oxidation of chromium to Cr+6 is not favorable or expected.

Additionally, the results of the reanalysis that are qualified as estimated values were similar to the rejected non-detect results of the initial analysis in that newly detected Cr+6 concentrations in the re-analysis were generally marginally above the respective reporting limit.

These results are considered to be usable in the context of the noted qualifications, in conjunction with other site information, with the results of the re-analysis being the more representative and recommended for reporting.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
*	Duplicate analysis not within control limits; indeterminate bias direction.
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.
R	The result is rejected because the MS recovery in the associated QC sample is below 50%;
NR	The matrix spike sample recovery in the associated QC sample is below the 50% QC limit recommending rejection of the result.
U	The analyte was analyzed for, but was not detected at the stated reporting limit.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

APHA, AWWA, and WEF, 1995, ***Standard Methods for the Examination of Water and Wastewater, 19th Edition***, Washington, D.C., 1268 p.

New Jersey Department of Environmental Protection, 2014a, ***Data of Known Quality Protocols Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014b, ***Data Quality Assessment and Data Usability Evaluation Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014c, ***Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2014d, ***Quality Assurance Project Plan Technical Guidance***, Version 1.0, Trenton, New Jersey, April 2014.

New Jersey Department of Environmental Protection, 2012, ***Technical Requirements for Site Remediation, N.J.A.C. 7:26E***, Trenton, New Jersey, May 7, 2012.

New Jersey Department of Environmental Protection, 2009, ***Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium, SOP No.: 5.A.10***, Trenton, New Jersey, September 2009.

New Jersey Department of Environmental Protection, 2002, ***Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16***, Trenton, New Jersey.

New Jersey Department of Environmental Protection, 2001, ***Standard Operating Procedure for the Analytical Data Validation of Target Analyte List - Inorganics BEMQA 5.A.2, Revision 4***, Trenton, New Jersey.

US EPA, CLP, 2017, ***“National Functional Guidelines for Inorganic Superfund Methods Data Review”***, OSWER Publication 9355.0-135, EPA-540-R-2017-001, January 2017.

US EPA, 2016, ***ICP-AES Data Validation, SOP HW-3a, Revision 1***, December 2016.

US EPA, 1997, ***Test Methods for Evaluating Solid Wastes, 3rd Edition including Final Update III***, Office of Solid Waste and Emergency Response, Washington, D.C., June 1997.

US EPA, 1992, ***Guidance for Data Usability in Risk Assessment (Part A) Final***, Office of Solid Waste and Emergency Response (OSWER), April 1992.

ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD17978

1. Were the appropriate sample preservation requirements met?..... **Yes** No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? **Yes** No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes **No**
Indicate the identity of the samples and why.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes **No**
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? Yes **No**
If "Yes", include the number of samples and laboratory sample ID numbers.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... **Yes** No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... **Yes** No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... Yes No
9. Were rejections noted in the non-conformance summary?..... Yes No
Provide a brief explanation.
10. Were rejected data used?..... Yes No
If “yes”, please indicate reasons rejected data were used:
 For Hex Chrome, data were rejected because spike recovery was <50%.
 Data were rejected due to missing deliverables.
 Data were rejected but an applicable standard exceedance exists.
 Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
 Other reasons not noted directly above. Explain:

Sample non-detect Cr+6 results were rejected in the initial analysis because the soluble MS recovery was below 50% and total chromium data was not available to determine potential usability of the data. Sample Cr+6 results in the reanalysis are qualified as estimated values, since the MS recovery was above 50%. It is recommended that the reanalysis Cr+6 results be used for reporting.

11. Were the quality control criteria associated with the compounds of concern at the site met? Yes No
12. Were the QC Summary Forms reviewed?..... Yes No
13. Internal Standards acceptable..... Yes No
14. MS/MSD acceptable..... Yes No
15. Calibration summaries acceptable..... Yes No
16. Serial dilutions acceptable..... Yes No
17. Inorganic duplicates acceptable..... Yes No
18. LCS recovery acceptable..... Yes No
19. Other QC acceptable?..... Yes No
20. Provide a brief explanation, if applicable.

Refer to DV report tables 2, 3, 4, 5, and 6 for QC details. Qualified sample results are presented in Table 7 of this DV report.



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DATA VALIDATION REPORT

Project: Jersey City PPG, Site 174; Report SDGs JD18044A
Sample Dates: December 21, 2020
Analyses: Metals Analysis, EPA Method 6010D
Percent Solids, SM2540 G 18th Ed. Mod.
Reviewer: Janis V. Giga, Ph.D., REP5554
Report Date: January 26, 2021

This data validation (DV) report presents the data review and result qualifications for two (2) soil samples collected at the PPG Site 174 (West First Street) in Bayonne, New Jersey on December 21, 2020 for sample delivery group (SDG) JD18044A. The samples were analyzed for the analytes listed above employing the identified analytical methods by SGS North America, Inc. Laboratories of Dayton, New Jersey.

Summary of Sample Results Qualifications

The soil sample analytical results for the samples of SDG JD18044A were found to be compliant with the analytical methods employed for the analysis of metals in the two collected soil samples.

Following the detailed DV review, the following sample results were qualified:

- Antimony ("NJ-") in Samples JD18044-1A and JD18044-2A.

No other sample results in SDG JD18044A required qualification, based on the acceptable remaining associated QC results and analytical performance. Details are provided in the tables and text below. The reported metals concentrations were below the respective Residential Soil Remediation Standard (SRS) and Impact to Groundwater Soil Screening Level (IGWSSL) limits, whichever was more stringent. A data validation checklist is provided in Attachment A to summarize the observations during the DV review.

The sample results that were subject to qualification following the DV review are presented in Table 3 of this DV report.

Sample Receipt

The two (2) soil samples collected December 21, 2020 received intact and appropriately preserved December 21, 2020 at the SGS laboratory in Dayton, NJ with acceptable sampling cooler temperatures with a maximum corrected temperature of 2.6°C. The field sample identification numbers and corresponding laboratory identification numbers are as follows:

Table 1. Sample Receipt Summary – SDG JD18044A

<u>Client Sample Designation</u>	<u>Sample Lab ID Number</u>	<u>Date Collected</u>	<u>Matrix</u>	<u>Analyses</u>
FNC-1A	JD18044-1A	12/21/2020	Soil	Metals
FNC-1B	JD18044-2A	12/21/2020	Soil	Metals
Metals – Antimony, chromium, nickel, thallium and vanadium analyzed by SW-846 Method 6010D at SGS Laboratories in Dayton, NJ, as well as percent total solids.				

The data package numbered JD18044A presents the metals data along with the total solids results for the two soil samples.

Data Review

Data, as presented in the analytical data packages SDG JD18044A was primarily reviewed and validated using the following combination of method-specific criteria with professional judgement, as appropriate:

- New Jersey Department of Environmental Protection (NJDEP) *Standard Operating Procedure: Quality Assurance Data Validation of Analytical Deliverables Inorganics (Based on USEPA SW-846 Methods), SOP No. 5.A.16* (NJDEP, 2002).
- United States (US) Environmental Protection Agency (EPA) *“National Functional Guidelines for Inorganic Superfund Methods Data Review”*, OSWER Publication 9335.0-135, EPA540-R-2017-001, January 2017 (US EPA, 2017).
- US EPA *“ICP-AES Data Validation, SOP No. HW-3a, Revision 1”* (US EPA, 2016).
- NJDEP *Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium* (NJDEP, 2009).
- NJDEP, *Data of Known Quality Protocols Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Data Quality Assessment and Data Usability Evaluation Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance*, Version 1.0, April 2014.
- NJDEP, *Quality Assurance Project Plan Technical Guidance*, Version 1.0, April 2014.

Data associated with parameters that do not meet quality control (QC) specifications or compliance requirements, have been qualified in accordance with US EPA Region II/NJDEP specifications/guidelines, as appropriate.

The analysis of the identified samples was performed in compliance with the requirements specified in the respective analytical methods. The data is presented in a NJDEP “reduced” deliverables package and is considered complete, as defined by the NJDEP “Technical Regulations for Site Remediation” (NJDEP, 2012). However, it is emphasized that due to the absence of raw metals data and the associated preparation logs, the substantiation of the reported metals concentrations and the accuracy of the QC summary results is precluded. The information presented in the data summary and quality control (QC) forms was reviewed and used to qualify the sample results. The quality of data collected in support of this sampling activity is considered acceptable with the noted results qualifications, considering the limitations attributable to a reduced deliverables data package.

The discussion below presents the findings of the data validation review organized according to the technical areas used to evaluate inorganic analytical data. For each of these analytical topics, the information on the summary forms, as well as the raw data and supporting information for the samples or standards analyzed were reviewed during the DV effort.

1.0 Metals Analysis Data Review – SDG JD18044A

The data validation of the metals analytical data in SDG JD18044A was reviewed for the following data quality items and a check mark (√) indicates successful achievement of meeting the relevant QC requirements:

- | | |
|---------------------------------|------------------------------|
| √ Holding times | Matrix spike recoveries |
| √ Blank Analysis | √ Duplicate analysis |
| √ Calibration standards | √ Laboratory control samples |
| √ Calibration verification | √ Serial dilution analysis |
| √ ICP Interference Check Sample | √ Data package completeness |
| √ Data qualifiers | |

The two soil samples were analyzed for the five target EPA Method 6010D metals (antimony, total chromium, nickel, thallium, and vanadium), as well as percent total solids, were covered by this data validation. Of the sample metals results detected in the 2 soil samples of SDG JD18044A, no results in the two samples exhibited a concentration above the IGWSSL or SRS, whichever was more stringent.

Laboratory Case Narrative

The case narrative stated that the matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were identified as being outside QC limits in QC Batch MP24497 indicating possible matrix interference and/or sample nonhomogeneity in the soil samples analyzed in this SDG. The case narrative also stated that the RPD serial dilution result for thallium was outside control limits in QC Batch MP24497, however, the percent difference (%D) result was acceptable due to a low initial sample concentration (< 50 times instrument detection limit [IDL]). No samples were diluted for the metals analysis. All other QC requirements for the analytes reviewed for data validation were met, including the analysis for total percent solids. Details are discussed in the sections below.

Holding times (QC Limit: 6 months)

The six-month analytical holding time was met for all inductively coupled plasma (ICP)-analyzed soil samples.

Calibration Standards (QC Limits: 90-110%; CRI QC Limit 70-130%)

The QC calibration requirements were met by the initial and continuing calibrations employed, including those of the high check standard and “low calibration check standard” (“CRI” standard), with target analyte recoveries all within the respective required QC limits, thereby demonstrating linearity for the soil sample analyses and acceptable analyte quantitation (concentration determination).

Hence, no sample results required qualifications for calibration issues.

Quality Control Blanks (QC Limit: < CRDL or <RL)

There were no target metals concentrations detected in the procedure blanks and the continuing calibration blanks (CCBs) at the stated reporting limit (RL) or contract required detection limit (CRDL).

No soil sample results warranted qualification for any associated QC blank contamination in SDG JD18044A.

ICP Interference Check Samples (QC Limits: 80-120%)

All analyte recoveries in the interference check samples, both IND A and IND B, were within the specified QC limits for the target compounds.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis
(QC Limits: 75-125% Recovery; ≤ 35%RPD)

The matrix spike (MS) and matrix spike duplicate (MSD) recoveries for antimony were below the QC limits of 75 - 125% for the non-client QC batch sample JD18289-1, as identified in Table 2 below. These recoveries indicate possible matrix interference and/or possible sample non-homogeneity. Following the DV review, the sample antimony results subject to qualification were flagged with “N” to indicate that the result is associated with a QC recovery outside QC limits and the antimony results further flagged with “J-” to indicate the possible presence of a potential low bias in the ability to recover antimony in the given sample matrix, in accordance with DV guidelines (USEPA, 2017; NJDEP, 2002). The remaining matrix spike results fell within QC limits in Batch MP24497.

Table 2. Matrix Spike Recovery Results Outside QC Limits

QC Batch	QC Sample	Analyte	MS Recovery	MSD Recovery	DV Qualifier	Potential Bias
MP24497 Ω	JD18289-1	Antimony	70.2 %	70.9 %	NJ-	Low
QC Limits are 75-125%; MS – Matrix spike MSD – Matrix spike duplicate. NJ- – The matrix spike recovery was below QC limits; associated sample results may experience a potential low bias. Ω – The samples associated with QC Batch MP24497 consist of JD18044-1A and JD18044-2A						

The antimony results in the two affected soil samples are flagged with “NJ-” due to a potential low bias. The qualified antimony results are presented below in summary table, Table 3.

Duplicate analysis (QC Limit: ≤ 35 %RPD)

The duplicate analysis was performed on one pair of spiked duplicate samples from non-client sample JD18289-1. All %RPD values for the 5 target analytes were below the laboratory QC limit of 20%RPD, as well as the project QC limit of 35%RPD for soil samples, with values ranging 0.0 – 6.1%RPD for soil samples with no results requiring qualification. The duplicate analyses demonstrated acceptable analytical precision.

Laboratory control samples (QC Limits: 80-120% Recovery)

All analyte recoveries in the laboratory control samples were within the specified QC limits demonstrating acceptable analytical system performance, with blank spike recoveries ranging 92.3% - 99.5% for the soil sample metals analysis.

Serial Dilution Analysis (QC Limit: ≤ 10 %D)

The case narrative stated that the RPD serial dilution result for thallium was outside control limits in QC Batch MP24497, however, the percent difference (%D) result was acceptable due to a low initial sample concentration (< 50 times IDL) of this analyte. The serial dilution results for the remaining four analytes associated with the soil samples ranged from 0.0 – 6.8%D, values below the QC limit of 10%D criterion for data validation qualification (US EPA, 2017).

No sample results required qualification for serial dilution issues.

Quantification Verification

Metals concentrations reported on the Form 1 sheets for the soil samples could not be verified because the data was provided in a NJDEP “Reduced deliverables” format (NJDEP, 2012), omitting the quantitation reports and preparation logs from the raw data.

Reporting Limits

No samples required dilution, such that all reporting limits were below the respective IGWSSL and SRS limit values.

Hence, all reporting limits were below the respective project IGWSSL and SRS limit values.

Summary of Qualified Metals Results

The soil sample analytical results for the samples of SDG JD18044A were found to be compliant with the analytical methods for the analysis of metals in the two soil samples using SW-846 Method 6010D.

The QC criteria were met for the ICP target analyte analyses, except for the low matrix spike recoveries for antimony in the QC batch associated with the 2 soil samples of this SDG, as detailed below in Table 3. The antimony results in these samples are qualified as estimated values (flagged “NJ-”) in the associated soil samples due to a potential low bias in the ability to recover antimony in the affected samples, as summarized below in Table 3.

Table 3. Summary of Qualified Sample Metals Results in SDG JD18044A

Sample ID	Lab ID	Analyte	Result (mg/kg)	DV Qualifier
FNC-1A	JD18044-1A	Antimony	< 2.3	NJ-
FNC-1B	JD18044-2A	Antimony	< 2.4	NJ-

Key:
 < –The analyte was analyzed for, but was not detected above the stated reporting limit.
 NJ- – The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

No other soil sample target metals results required qualification for any associated QC issues following the DV review.

The reported sample results are usable within the context of the applied qualifications, based on data usability considerations.

3.0 DATA QUALIFIER DEFINITIONS

The absence of qualifiers indicates that the data are acceptable both qualitatively and quantitatively.

Qualifier	Definition
J	The reported result is an estimated value.
N	The matrix spike sample recovery in the associated QC sample is not within QC limits.
NJ-	The matrix spike sample recovery in the associated QC sample is below QC limits; the result is estimated and may be biased low.

4.0 References

AECOM, 2010, ***Field Sampling Plan / Quality Assurance Project Plan for Non-Residential and Residential Chromium Sites, Hudson County, New Jersey***, dated June 2010.

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ATTACHMENT A

Data Validation Checklist

DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST

Project: PPG SDGs: JD18044A

1. Were the appropriate sample preservation requirements met?..... **Yes** No

2. Were appropriate sample holding times
(for both extraction/sample preparation and analysis) met? **Yes** No
If "No", provide a brief explanation.

3. Were the samples diluted? Yes **No**
Indicate the identity of the samples and why.

4. If applicable, did sample dilutions result in elevated reporting limits that exceed applicable standards?..... Yes **No**
If "Yes", list the affected samples.

5. Were any applicable standards exceeded for any samples? Yes **No**
If "Yes", include the number of samples and laboratory sample ID numbers.

6. Were the laboratory reporting limits below the applicable remediation standards/criteria required for the site?..... **Yes** No
If "No", provide a brief explanation of action taken.

7. Were qualifications noted in the non-conformance summary?..... **Yes** No
Provide a brief explanation.

Refer to DV report discussions of case narrative regarding QC limit exceedances. No problems with analytical procedures were noted.

8. Were qualified data used?..... **Yes** No

9. Were rejections noted in the non-conformance summary?..... Yes **No**
Provide a brief explanation.

Not applicable

10. Were rejected data used?..... Yes **No**

If "yes", please indicate reasons rejected data were used:

- For Hex Chrome, data were rejected because spike recovery was <50%.
- Data were rejected due to missing deliverables.
- Data were rejected but an applicable standard exceedance exists.
- Data were rejected in an early phase of remediation; however, additional sampling and analysis are scheduled to be performed.
- Other reasons not noted directly above. Explain:

11. Were the quality control criteria associated with the compounds
of concern at the site met? Yes **No**

12. Were the QC Summary Forms reviewed?..... **Yes** No

13. Internal Standards acceptable..... **Yes** No

14. MS/MSD acceptable..... Yes **No**

15. Calibration summaries acceptable..... **Yes** No

16. Serial dilutions acceptable..... **Yes** No

17. Inorganic duplicates acceptable..... **Yes** No

18. LCS recovery acceptable..... **Yes** No

19. Other QC acceptable?..... **Yes** No

20. Provide a brief explanation, if applicable.

Refer to DV report table 2 for QC details. Qualified sample results are presented in Table 3 of this DV report.