

Data Usability Report 3 Cleanup Verification Sampling Data

Krejci Dump Site

**Cuyahoga Valley National Park
Summit County, Ohio
May 2012**

The National Park Service



**Prepared by:
MCG Geotechnical Engineering, Inc.
4817 South Zang Way
Morrison Colorado 80465
Phone: (303) 973-2660**

TABLE OF CONTENTS

Section 1. Introduction	1
Section 2. Data Usability Methods and Results	2
2.1 Calculation of TEQ.....	2
2.2 Field Oversight	4
2.3 Laboratory Oversight	4
2.4 QC Measurements	5
2.5 Laboratory QC	6
2.6 Field QC	17
2.7 Data Qualifiers	22
2.8 Reliance Level.....	23
Section 3. Data Usability Summary.....	27
3.1 Dioxin and Furans	27
3.2 West Site Grid B03	31
Section 4. RG Achievement.....	31
Section 5. Conclusion	33
Section 6 Related References	34

Tables

Table 2.1 Dioxin and Furan Congeners and Associated TEFs.....	3
Table 2.2 Laboratory QC associated with sample WS-B03-110825 from database and laboratory report .	7
Table 2.3 Results of Test on 14 Duplicate CRM Sets ("Made to" Concentration was 3 pg/g)	18
Table 2.4 Result of Tests on Dioxin/Furan CRMs Submitted from the Field.....	20
Table 2.5 Statistics Related to Tests on Dioxin/Furan CRMs Submitted from the Field	20
Table 2.6 Results of Dioxin/Furan Measurements for Duplicate Splits	25
Table 2.7 RPD's Calculated for Duplicate Splits	26
Table 2.8 Inorganic Analyte RGs, RL's, and WS-B05-110825 Results.....	27
Table 3.1 Grids and Dioxin Sampling Areas having Calculated TEQ's Less than the RG but Greater than the RL	28

FIGURES

Figure 2.1 Dioxin and Furan LCS Recoveries (percent)	12
Figure 2.3 Dioxin and Furan LCSD RPDs	12
Figure 2.5 Dioxin and Furan Matrix Spike Recovery (percent)	13
Figure 2.7 Dioxin and Furan Matrix Spike Duplicate RPDs.....	14

APPENDICES

Appendix A. CVS and Comparisons to Remediation Goals
--

LIST OF ACRONYMS

AOC	Area of Concern
CD	Consent Decree
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CVS	Cleanup Verification Sampling
CV	Coefficient of Variation
CVSP	Cleanup Verification Sampling Plan
DQE	Data Quality Evaluation
DQO	Data Quality Objective
DUR	Data Usability Report
EQIS	EQ Industrial Services, Inc.
FSP	Field Sampling Plan
FQAO	Field Quality Assurance Officer
Ford	Ford Motor Company
LPC	Laboratory Performance Criteria
LOQ	Limit of Quantitation
MQO	Measurement Quality Objective
OSR	National Park Service On-Site Representative
OU	Operable Unit
Park	Cuyahoga Valley National Park
PID	Photoionization Detector
PQL	Practical Quantitation Limit
QA	Quality Assurance
QAP	Quality Assurance Plan (Laboratory)
QAPP	Quality Assurance Project Plan
QC	Quality Control
RA	Remedial Action
RI	Remedial Investigation
RAWP	Remedial Action Work Plan
RD	Remedial Design
RG	Remediation Goal
RL	Reliance Level
RPD	Relative Percent Difference
ROD	Record of Decision
Site	Krejci Dump Site
SOW	Statement of Work
TEQ	2,3,7,8 TCDD Toxicity Equivalent
VOC	Volatile Organic Compound

Section 1. Introduction

This Krejci Dump Site (Site) Data Usability Report (DUR) 3 was commissioned by NPS to enable NPS's independent evaluation and determination of: (1) whether the Cleanup Verification Sampling (CVS) data for the Site are of sufficient quality for use in determining achievement of Site Remediation Goals (RGs); and (2) whether the usable data establish that the RGs have been achieved. The usability evaluation of CVS data is set forth in three DURs. DUR 1 evaluates CVS data for soil samples collected at the end of initial excavation and that were reported in the September 11, 2009 CVS Database. DUR 2 evaluates the CVS data first reported in the December 16, 2010 CVS Database (i.e., entered after September 11, 2009), excluding dioxin/furan data. This document, DUR 3, evaluates all remaining CVS test results. This includes an evaluation of CVS data for West Site grid B03 (sample WS-B03-110825) that is first reported in the October 7, 2011 CVS database, as well as all dioxin/furan data (including dioxin/ furan data preliminarily addressed in DUR 1). DUR 1 introductory Sections 2 – 6 (explaining, e.g., the data quality objectives (DQOs) and data quality assurance program) are pertinent to this DUR and incorporated herein by reference.

The data usability assessment proceeds as follows: (1) all CVS measurements are compared to project measurement quality objectives (MQOs), since measurements that attain all MQOs are usable; (2) all CVS measurements are then compared to a derived reliance level (RL), which is a calculated concentration that sets a limit on how close a CVS measurement can be to the RG without undue concern that noncompliance with MQOs might impact decisionmaking; and (3) CVS measurements that exceed the reliance level and do not achieve all MQOs are then individually evaluated using other contextual factors such as other related CVS data (e.g., other CVS results for same analyte in the same grid, CVS results for other analytes in the same analyte group in the same grid, CVS results for linked or associated analytes in the same grid), field and laboratory batch-specific data quality indicators, and qualitative data collection proficiency measures, to determine if the acquired measurement quality is sufficient to support the RG achievement decision.

This report is organized as follows: Section 2 evaluates data laboratory performance criteria (LPC) and MQO compliance; Section 3 completes the data usability evaluation for remaining dioxin/furan measurements and West Site Grid B03; Section 4 compares usable data to RGs; and Section 5 presents a summary of data usability and RG attainment. Appendix A includes a table containing all dioxin and furan test results organized by location and date (sample identifier) and compares them to RGs. Shaded columns in Appendix A graphically present the time of occurrence and the approximate depth of excavation for excavation events in response to dioxin and furan RG failures that were not previously presented in DUR 2 Appendix B. All October 7, 2011 dioxin and furan database entries relevant to usability and RG achievement decisions, including East Site dioxin/furan data previously presented in DUR 1, is presented in Appendix A.

DURs 1 and 2 concluded that the CVS data representing conditions that followed excavations to remove contaminants and that is contained in the September 11, 2009 and December 16, 2010 databases,

respectively, does have the quality necessary to use in making decisions regarding the attainment of RGs. DUR 1 compared this usable CVS data to the RGs and identified RG achievement by analyte, parameter group, and Site area (grid). Out of a total 186 Site grids, DUR 1 established that 69 grids met all RGs for all applicable analytes at the conclusion of the initial Site excavation in June, 2007. Grids that did not achieve all RGs after the initial Site excavation underwent additional rounds of excavation followed by CVS until the CVS results indicated RG achievement. DUR 2 concluded that 185 of the 186 Site grids met all RGs other than the RG for dioxins/furans, for which the data was not evaluated. West Site grid B3 (WS-B3) was the single grid excluded from the above conclusion because it remained in use as the West Site staging area. After removal of the staging area and in response to the chromium RG exceedance determined in DUR 1, WS-B3 underwent excavation followed by additional CVS of all analytes in the metals parameter group. Dioxin/furan and recent WS-B3 CVS results are evaluated herein and compared to respective RGs.

This data usability assessment evaluates the data as contained in the October 7, 2011 CVS database, which, prior to entry into the database, underwent laboratory verification and independent validation. Data quality evaluation was then undertaken by EQIS as documented in Data Quality Evaluation reports DQE 1, DQE 2 and DQE 3. The October 7, 2011 CVS database includes all CVS data, except: (i) it lacks inclusion of data qualifiers, which will be added to the final version of the database; and (ii) it does not include the results of tests performed on 14 duplicate sets of certified reference standards that were performed in 2005 at the beginning of excavation to evaluate dioxin/furan analytical accuracy and precision. This data is evaluated herein and will be added to the final database.

Section 2. Data Usability Methods and Results

2.1 Calculation of TEQ

Dioxins and furans are separate families of chemicals with similar molecular structures. Molecules that have the same basic structure (same family) but different formulas are called congeners. In all, there exist 210 congeners of dioxins and furans, each with varying toxicity. The congener of greatest environmental concern is 2,3,7,8 tetrachlorodibenzo-par-dioxin (2,3,7,8 TCDD). Although 2,3,7,8 TCDD is relatively rare, it was found on Site along with other congeners at unusually high concentrations during the Remedial Investigation (RI). To avoid the need for a remediation goal for each congener, dioxins and furans are often considered collectively as a Toxicity Equivalence (TEQ). Each dioxin-like congener having an established level of toxicity is assigned a Toxicity Equivalency Factor (TEF) based on its toxicity relative to 2,3,7,8 TCDD (assigned TEF of 1.0). The TEQ is then calculated as the sum of the products of congener concentrations and their respective TEFs. The TEF procedure was developed under the auspices of the North Atlantic Treaty Organization's Committee on Challenges of Modern Society to promote international consistency in addressing dioxin/furan contamination, and the TEFs as

defined in I-TEFs/89 for the fourteen (14) identified Site congeners are presented in Table 2.1.¹ Often a TEQ calculation includes seventeen (17) dioxin/furan congeners. For calculation of the dioxin/furan TEQ RG at the Krejci Site, however, three common congeners (OCDD, 2,3,7,8 TCDF, and 1,2,3,4,6,7,8-HpCDD) were excluded from the TEQ RG calculation because they were found in field or laboratory blank samples and, therefore, the measured concentrations could not be definitively associated with the Site. (See Section 2.5 herein for additional discussion.) The RG for the Krejci Site, 3 pg/g, is a TEQ derived using the RI data for the background 14 congeners identified in Table 2.1.

Table 2.1 Dioxin and Furan Congeners and Associated TEFs

Congener	TEF
1,2,3,4,6,7,8-HpCDF	0.01
1,2,3,4,7,8,9-HpCDF	0.01
1,2,3,4,7,8-HxCDD	0.1
1,2,3,4,7,8-HxCDF	0.1
1,2,3,6,7,8-HxCDD	0.1
1,2,3,6,7,8-HxCDF	0.1
1,2,3,7,8,9-HxCDD	0.1
1,2,3,7,8,9-HxCDF	0.1
1,2,3,7,8-PeCDD	0.5
1,2,3,7,8-PeCDF	0.05
2,3,4,6,7,8-HxCDF	0.1
2,3,4,7,8-PeCDF	0.5
2,3,7,8-TCDD	1
OCDF	0.001

We note that all seventeen dioxin/furan congeners were measured and presented in the October 7, 2011 database and related data summaries; this is because the laboratory analyzed and reported the congener data as a set. Only the 14 above-identified congeners, however, were used for calculating the

¹ North Atlantic Treaty Organization's Committee on Challenges to Modern Society (NATO/CCMS), 1988. *International Toxicity Equivalency Factor (I-TEF) Method of Risk Assessment for Complex Mixtures of dioxins and Related Compounds*. Report No. 176. As referenced in the United States Environmental Protection Agency (USEPA) 1994. *Estimating Exposure to Dioxin-Like Compounds*. EPA/600/6-88/005 Ca. June 1994.

dioxin/furan TEQ for the purpose of determining achievement of the dioxin/furan RG, so only the CVS and QC data for these 14 congeners are reviewed and evaluated herein.

2.2 Field Oversight

Many tasks performed on Site during remedial action (RA) activities have the potential to significantly influence the quality of the cleanup and the confidence with which decisions may be made regarding attainment of RGs. Procedures for those tasks considered most influential are detailed in the Remedial Design (RD) Report and Remedial Action Work Plan (RAWP), such that it is likely that a remediation activity was successful when the prescribed procedures were performed as specified. Two general approaches were used to assure that adequate performance of field procedures was achieved. First to the extent practical, measurements were made and compared to project specific acceptable standards of practice, such as MQOs and laboratory performance criteria (LPCs). Second, when measurements were not applicable or practical, compliance with procedures was assured by close monitoring of activities (field and laboratory oversight). Such monitoring allowed early detection of procedural errors and immediate development and implementation of corrective actions.

The EQIS Project Manager (PM), Quality Assurance Officer (QAO), Field Quality Assurance Officer (FQAO) and Site personnel were responsible for assuring that procedures and practices were implemented according to the RD Report and RAWP. Additionally, NPS's On-Site Representative (OSR) was present during all RA field activities and provided an independent level of review. NPS also maintained an off Site group of technical experts with whom the OSR communicated on a regular basis, who reviewed all documents and plans related to the implementation of the RAWP, and who were available for consultation with the OSR whenever questions arose. The FQAO and the OSR prepared independent daily reports of activities and observations. These reports documented all Site activities, problems encountered and corrective actions taken. The OSR reports were reviewed by the NPS technical experts to evaluate compliance with the RA and RAWP; identify potential problems and propose corrective actions; and evaluate the impact of problems and corrective actions on the quality of the RA and related decisions. By this process field problems were identified and corrected immediately, thereby reasonably assuring procedures were implemented as expected and a high quality cleanup resulted. Furthermore, the process assured that CVS collection activities were in accordance with the Field Sampling Plan (RD Report, Appendix C), thereby assuring that errors did not occur during this effort and that CVS of acceptable quality were collected for testing.

2.3 Laboratory Oversight

The dioxin/furan CVS soil samples were sent from the Site to CT Laboratories, Baraboo, Wisconsin, where they were ground, homogenized, and split according the FSP procedure prior to being mailed to the Test America Laboratory in Sacramento, California for dioxin/furan analyses. Preparation and analysis of sample WS-B03-110825 (the only sample other than dioxin/furan samples that was not evaluated in previous DURs) were both performed at CT Laboratories.

Compliance with industry accepted standards of practice with regard to specified analytical procedures was assured by evaluating the laboratory quality assurance plan (QAP) and ensuring its implementation. For some analytical methods this project required attainment of data quality that exceeded common practice because the decisions regarding attainment of RGs for a grid are based on analyses of one multi-increment sample. Project specific standards are presented as LPCs in the QAPP, Table 2.3, and are the focus of comparisons presented in DUR 1 and DUR 2 for all chemicals other than dioxin/furan. LPCs for dioxin/furan analyses are presented and discussed herein and compared to quality control measures.

NPS technical experts audited testing laboratories and reviewed and approved the QAPs and operating procedures prior to the start of laboratory analyses of CVS. A laboratory quality assurance officer monitored day-to-day operations within the laboratory and immediately corrected problems that could adversely impact data quality. The EQIS quality assurance officer (QAO) operated independently of the laboratory and provided oversight that included laboratory audits and data validation in accordance with QAPP specified procedures. Data validation reports and audits were documented by the QAO and reviewed by NPS technical experts. Additionally, NPS retained a technical expert to provide independent oversight of laboratory audits and to spot check laboratory and validation reports. By this process, laboratory problems were identified and immediately corrected, thereby reasonably assuring that procedures were implemented as expected and that CVS measurements of acceptable quality were made.

2.4 QC Measurements

Quality control (QC) measurements were made during the RA to evaluate attainment of MQOs and LPCs. Samples were submitted by NPS, EQIS, and the laboratory for this purpose. QC protocols and criteria were implemented with various methods and procedures to evaluate measurement systems and to demonstrate that data of known quality were generated. QC protocols and criteria and their interpretation are included in the QAPP. Consistent with the Consent Decree (CD), a CVS measurement that meets QAPP-specified LPCs and MQOs is acceptable for making project related decisions without qualification. A measurement that does not meet QAPP-specified LPCs or MQOs may be used with qualification if, by subsequent data usability evaluation, it is demonstrated to be of sufficient quality to permit decisions to be made with acceptable confidence. [Acceptable decisionmaking confidence is discussed in DUR 1 Sections 3 and 7.]

QC measurements needed to evaluate achievement of MQOs and some of the QC measurements needed to evaluate achievement of LPCs are presented in the October 7, 2011 database for each dioxin/furan congener. Additionally, results of 28 repeated dioxin/furan analyses of a certified reference material and the associated data validation report, transmitted by letter dated August 3, 2006 to Mr. Robert McCaig, NPS Project Coordinator, from Mr. Bernd Rehm, EQIS Project Manager, are significant to demonstrating data quality and are summarized and discussed in Section 2.6. QC measurements associated with West Site grid B3 CVS for metals are also evaluated in Section 2.6. DQE's 1, 2 and 3 also evaluate MQO and LPC compliance and assign qualifiers to noncompliant data.

Additionally, laboratory and field QC are described, evaluated and discussed throughout the remainder of this report.

2.5 Laboratory QC

Many different types of tests and checks were made by the laboratory to assure data quality. The database contains the following dioxin/furan laboratory analysis QC results: sample duplicate (SD); laboratory control (LC); laboratory control duplicate (LCD); matrix spike (MS); matrix spike duplicate (MSD); and laboratory blank (B) samples. Also, the database includes sample collection dates, sample extraction dates, and sample test dates that are used to calculate sample holding times to extraction and analysis. Each data type is discussed in the following paragraphs. Laboratory quality control measures pertinent to metals analyses are discussed and presented in DUR 1, Section 7 and are incorporated herein by reference. This data, pertinent to dioxin/furan analyses and metals analyses for West Site Grid B03, are presented graphically in Table 2.2 and discussed in the remainder of this Section.

Homogenization Success Determination by KNO₃ Spiking

Potassium Nitrate, KNO₃ was added to each sample as it was received in the laboratory (aka “spiking” the sample) but prior to homogenization to create a heterogeneous condition that successful homogenization was expected to remove. The MQO in the QAPP was to assure the RPD of duplicate NO₃⁻² measurements are always less than 35 percent following homogenization. Duplicate NO₃⁻² measurements were made for each of 460 homogenized samples and the MQO was achieved with one exception, which had a small exceedance (RPD = 36 percent).

Dioxins/Furans

Analyses of duplicate subsamples spiked with KNO₃ afforded a measure of homogenization success. However, to avoid the possible introduction of low level dioxins or furans to samples, KNO₃ was not added to dioxin/furan samples. DURs 1 and 2, Sections 7.4 and 2.4 respectively, concluded that the homogenization process was successful and did not greatly influence measurement quality. Based on these findings and because samples for dioxin/furan analyses were prepared by the same homogenization process and by the same laboratory used to prepare all previous samples, it is considered unlikely that homogenization inefficiencies significantly influence dioxin/furan measurement quality.

West Site Grid B03

KNO₃ was spiked into the West site CVS sample for grid B03, WS-B03-110825, and the RPD calculated from tests performed on two splits created following grinding and homogenization was 34 percent, thereby achieving the measurement quality objective of 35 percent. Good past performance and a demonstrated RPD less than 35 percent suggest that homogenization efficiency does not significantly influence WS-B03-110825 measurement quality.

Table 2.2 Laboratory QC associated with sample WS-B03-110825 from database and laboratory report

Test Batch	Analyte	Sample Identifier	Sample Type	Recovery	RPD
78728	BORON	968332	LCS	92	2
78728	BORON	968333	LCSD	94	
78728	BORON	WS-B03-110825DUP	Laboratory Duplicate		15
78728	BORON	WS-B03-110825MS	MS	81	2
78728	BORON	WS-B03-110825MSD	MSD	79	
78728	MOLYBDENUM	968332	LCS	103	4
78728	MOLYBDENUM	968333	LCSD	107	
78728	MOLYBDENUM	WS-B03-110825DUP	Laboratory Duplicate		2
78728	MOLYBDENUM	WS-B03-110825MS	MS	89	3
78728	MOLYBDENUM	WS-B03-110825MSD	MSD	92	
78729	ALUMINUM	968338	LCS	109	16
78729	ALUMINUM	968339	LCSD	93	
78729	ALUMINUM	WS-B03-110825DUP	Laboratory Duplicate		0
78729	ALUMINUM	WS-B03-110825MS	MS		
78729	ALUMINUM	WS-B03-110825MSD	MSD		
78729	CHROMIUM	968338	LCS	120	0
78729	CHROMIUM	968339	LCSD	120	
78729	CHROMIUM	WS-B03-110825DUP	Laboratory Duplicate		1
78729	CHROMIUM	WS-B03-110825MS	MS	102	5
78729	CHROMIUM	WS-B03-110825MSD	MSD	78	
78729	LEAD	968338	LCS	105	1
78729	LEAD	968339	LCSD	106	
78729	LEAD	WS-B03-110825DUP	Laboratory Duplicate		1
78729	LEAD	WS-B03-110825MS	MS	84	1
78729	LEAD	WS-B03-110825MSD	MSD	83	
78729	VANADIUM	968338	LCS	119	1
78729	VANADIUM	968339	LCSD	120	
78729	VANADIUM	WS-B03-110825DUP	Laboratory Duplicate		8
78729	VANADIUM	WS-B03-110825MS	MS	92	3
78729	VANADIUM	WS-B03-110825MSD	MSD	88	
78731	CADMIUM	968332	LCS	100	0
78731	CADMIUM	968333	LCSD	100	
78731	CADMIUM	WS-B03-110825DUP	Laboratory Duplicate		<MDL
78731	CADMIUM	WS-B03-110825MS	MS	110	4
78731	CADMIUM	WS-B03-110825MSD	MSD	100	
78731	COBALT	968332	LCS	106	4
78731	COBALT	968333	LCSD	110	

Test Batch	Analyte	Sample Identifier	Sample Type	Recovery	RPD
78731	COBALT	WS-B03-110825DUP	Laboratory Duplicate		4
78731	COBALT	WS-B03-110825MS	MS	86	0
78731	COBALT	WS-B03-110825MSD	MSD	86	
78731	MANGANESE	968332	LCS	106	3
78731	MANGANESE	968333	LCSD	109	
78731	MANGANESE	WS-B03-110825DUP	Laboratory Duplicate		3
78731	MANGANESE	WS-B03-110825MS	MS		
78731	MANGANESE	WS-B03-110825MSD	MSD		
78731	SILVER	968332	LCS	94	1
78731	SILVER	968333	LCSD	95	
78731	SILVER	WS-B03-110825DUP	Laboratory Duplicate		0
78731	SILVER	WS-B03-110825MS	MS	88	1
78731	SILVER	WS-B03-110825MSD	MSD	90	
78732	BARIUM	968326	LCS	110	0
78732	BARIUM	968327	LCSD	110	
78732	BARIUM	WS-B03-110825DUP	Laboratory Duplicate		2
78732	BARIUM	WS-B03-110825MS	MS	117	1
78732	BARIUM	WS-B03-110825MSD	MSD	115	
78732	BERYLLIUM	968326	LCS	110	0
78732	BERYLLIUM	968327	LCSD	110	
78732	BERYLLIUM	WS-B03-110825DUP	Laboratory Duplicate		2
78732	BERYLLIUM	WS-B03-110825MS	MS	105	1
78732	BERYLLIUM	WS-B03-110825MSD	MSD	105	
78732	COPPER	968326	LCS	90	2
78732	COPPER	968327	LCSD	92	
78732	COPPER	WS-B03-110825DUP	Laboratory Duplicate		2
78732	COPPER	WS-B03-110825MS	MS	120	1
78732	COPPER	WS-B03-110825MSD	MSD	116	
78732	NICKEL	968326	LCS	109	1
78732	NICKEL	968327	LCSD	110	
78732	NICKEL	WS-B03-110825DUP	Laboratory Duplicate		0
78732	NICKEL	WS-B03-110825MS	MS	94	1
78732	NICKEL	WS-B03-110825MSD	MSD	98	
78732	ZINC	968326	LCS	104	3
78732	ZINC	968327	LCSD	107	
78732	ZINC	WS-B03-110825DUP	Laboratory Duplicate		1
78732	ZINC	WS-B03-110825MS	MS	106	1
78732	ZINC	WS-B03-110825MSD	MSD	112	

Test Batch	Analyte	Sample Identifier	Sample Type	Recovery	RPD
78813	MERCURY	970877	LCS	102	
78813	MERCURY	WS-B03-110825MS	MS	102	1
78813	MERCURY	WS-B03-110825MSD	MSD	105	
78798	ARSENIC	968344	LCS	115	1
78798	ARSENIC	968345	LCSD	116	
78798	ARSENIC	WS-B03-110825DUP	Laboratory Duplicate		0
78798	ARSENIC	WS-B03-110825MS	MS	102	3
78798	ARSENIC	WS-B03-110825MSD	MSD	106	
78757	SELENIUM	968344	LCS	103	6
78757	SELENIUM	968345	LCSD	97	
78757	SELENIUM	WS-B03-110825DUP	Laboratory Duplicate		
78757	SELENIUM	WS-B03-110825MS	MS	87	9
78757	SELENIUM	WS-B03-110825MSD	MSD	92	
38429	ANTIMONY	968347	LCS	110	1
38429	ANTIMONY	968348	LCSD	109	
38429	ANTIMONY	WS-B03-110825DUP	Laboratory Duplicate		7
38429	ANTIMONY	968347	MS	56	3
38429	ANTIMONY	968348	MSD	61	

Laboratory Blanks

Dioxins/Furans

A powdered sodium sulfate (Na_2SO_4) sample was processed and analyzed as a laboratory blank with each sample batch. The furan congener 2,3,7,8 TCDF and the dioxin congener OCDD were the only congeners detected in the blanks. These same two congeners, along with 1,2,3,4,6,7,8-HpCDD, were also detected in blanks analyzed during the remedial investigation (RI) and consequently were not included in RI calculations that ultimately resulted in the dioxin/furan TEQ RG. It is concluded, therefore, that OCDD and 2,3,7,8 TCDF are laboratory contaminants. The potential presence of these congeners will not impact data usability or comparability, because these congeners were excluded from both the TEQ used to derive the RG and the CVS TEQ calculated to evaluate RG achievement.

Confidence regarding comparability is gained by 1) noting that the same laboratory, Test America (Sacramento), was used to analyze both RI and CVS dioxin/furan samples; and 2) a comparison of the Test America SW846 8290 dioxin/furan laboratory procedures used for RI sample analyses and later for CVS analyses (as documented in audit reports) revealed only minor, inconsequential procedural changes.

West Site Grid B03

The database does not contain laboratory blank measurements, however, the laboratory report presenting the results of grid B03 metals analyses states that contaminants were not detected in the laboratory blanks. It is concluded, therefore, that laboratory contaminants are not expected to impact data usability.

Holding Times

Sample holding times have importance to data quality in that the risk of concentration-altering events, such as chemical reactions, biodegradation, photoionization, volatilization, chemical adsorption to glass, and loss or contamination due to leaky seals, increases with time. DUR 1 includes a discussion about the general impacts and significance of holding time exceedance on data usability, which is incorporated herein by reference. QAPP Table 3.2 establishes project specific holding times for analyte groups. Compliance with holding times for the first set of samples collected following initial excavation is discussed DQE 1, Section 7.4. Holding times for subsequent analyses other than dioxin and furan CVS and WS-B03-110825 are presented in DUR 2, Section 2.4.

Dioxins and Furans

The QAPP specified maximum holding times for dioxins/furans analyses are 30 days between sample collection and extraction and 45 days between extraction and analysis. Only one sample, WS-H04-080613, exceeded the 30 day holding time to extraction. No samples exceeded the 45 day holding time to analysis. The holding time exceedance for sample WS-H04-080613 has no impact on any RG decisionmaking, however, because the grid measured a dioxin/furan RG exceedance and underwent subsequent excavation and CVS.

West Site Grid B03

The QAPP-specified maximum holding time for inorganic analytes is 180 days from sample collection through and analysis, except for mercury that may be held for only 28 days from sample collection through analysis. Analyses for WS-B03-110825 were performed within these holding times.

Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control sample duplicates (LCSD) are samples of known concentration analyzed with each test batch (of about 20 samples) to provide a measure of accuracy and precision.

Dioxins and Furans

LCS and LCSD analyses were performed and the calculated RPD provides a measure of instrument measurement precision, among other things. For dioxins and furans, the LCS is created by spiking a blank Na₂SO₄ sample with each of the target 17 congeners and processing the sample as a CVS sample. LCSs were monitored closely by the laboratory and procedural or instrument corrections made as needed to assure acceptable analytical measurements. LCS percent recoveries of 17 congeners representing 15 batches are graphically presented on Figure 2.1. The median recovery is indicated by a

vertical line inside the shaded box; the horizontal limits of the box represent the estimated 25th and 75th percentiles; and the whiskers extend to the estimated 10th and 90th percentiles. For each congener, the outlying maximum and minimum recoveries are indicated by black dots when the value is outside the calculated 10th to 90th percentile range. Less than 1 percent of the 210 calculated recoveries were below 80 percent; also, the median recovery for each congener was as greater than 95 percent. These statistics represent overall good laboratory measurement accuracy.

Figure 2.2 presents the relative percent difference (RPD) for duplicate LCS measurements. Note that the horizontal axis on this plot is a logarithmic scale. For each congener, median recovery is indicated by a vertical line inside the shaded box; the horizontal limits of the box represent the estimated 25th and 75th percentiles; and the whiskers extend to the estimated 10th and 90th percentiles. The outlying maximum and minimum RPDs are presented as black dots on the figure when the value is outside the calculated 10th to 90th percentile range. Only one RPD exceeds 20 percent and the median RPDs for all congeners are less than 6 percent. These statistics indicate generally good analytical precision.

West Site Grid B03

Calculated recovery and RPD of LCS and LCSD for WS-B03-110825 are presented in Table 2.2 and exhibit recoveries between 90 and 120 percent and RPDs are between 0 and 16 percent. These ranges are within the limits of the QAPP-specified laboratory control limits of 80 and 120 percent for recovery and 20 percent for RPD. Past LCS and LCSD performance is presented in DUR 2, Section 2.4 and provides insight regarding potential intermittent errors and the potential effect of such errors on data usability. The cause of intermittent errors was not discovered and, therefore, no corrective action was taken and it was assumed that similar intermittent errors may affect the analysis of WS-B03-110825. Suffice it to say that intermittent errors are incorporated in the calculation of reliance level and considered in the evaluation of WS-B03-110825 data usability as presented in Section 2.8.

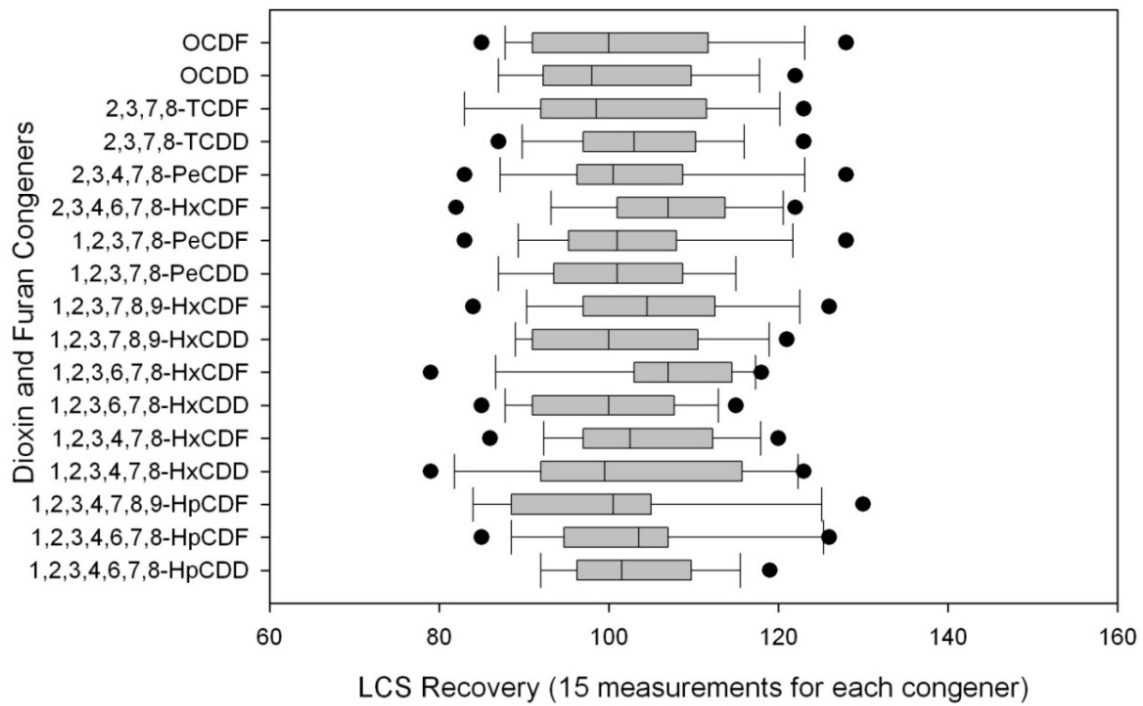


Figure 2.1 Dioxin and Furan LCS Recoveries (percent)

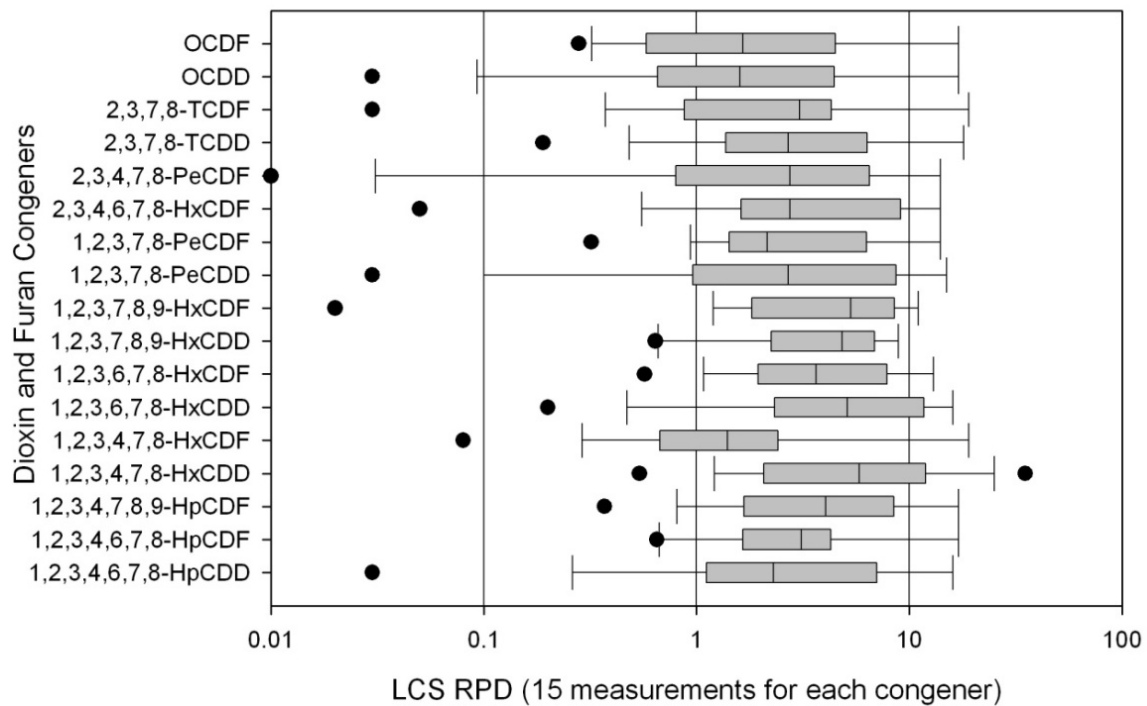


Figure 2.2 Dioxin and Furan LCSD RPDs

Matrix Spike and Matrix Spike Duplicates

A matrix spike (MS) is a CVS sample to which a known quantity of a target parameter is added. A matrix spike sample is similar to an LCS except the target congeners are spiked into a CVS rather than an inert blank granular material. The difference between laboratory measurement performance described by recoveries and RPDs of the LCS and the analogous measurements of the matrix spike distinguishes the effect of the CVS soil matrix on analyte identification and quantification.

Dioxins and Furans

A matrix spike sample was analyzed with every batch of dioxin/furan samples tested. Recovery and RPDs express as percentages are presented as Box-and-Whiskers plots on Figures 2.3 and 2.4, respectively.

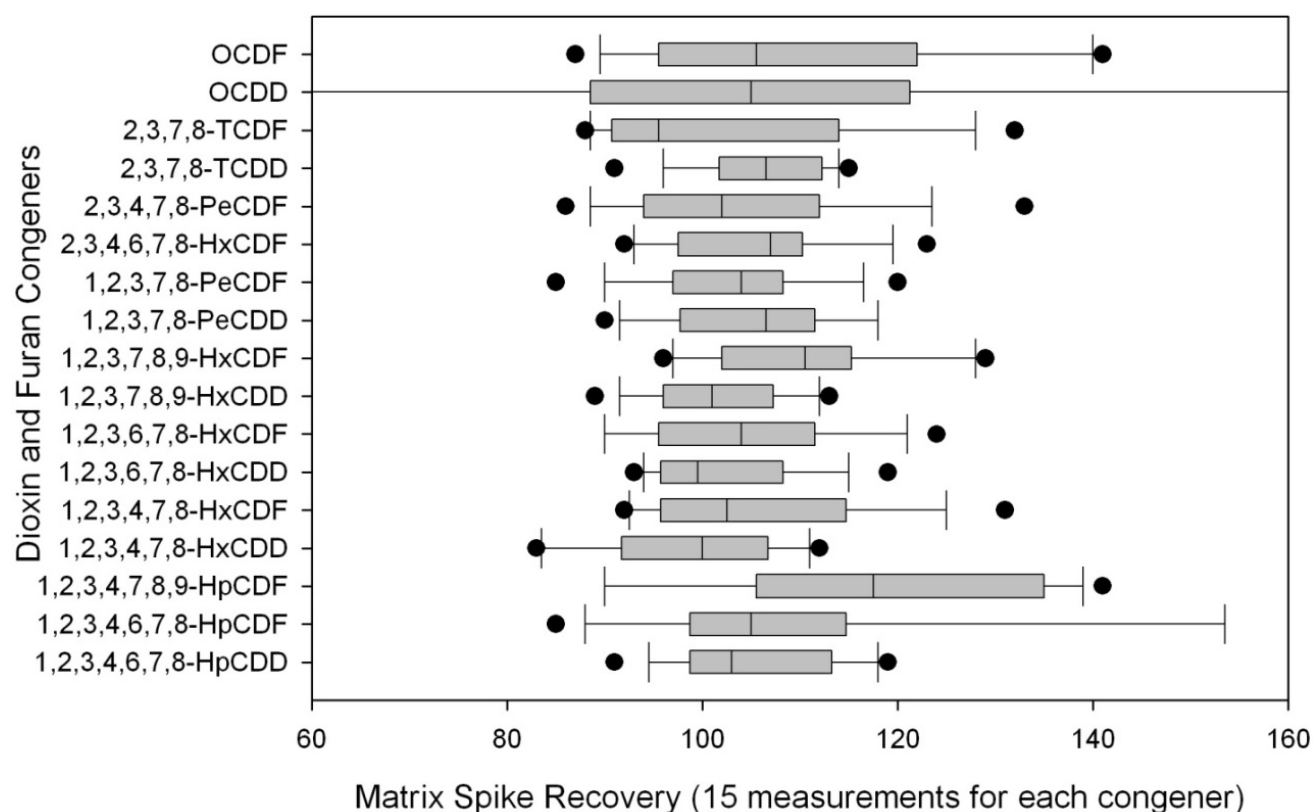


Figure 2.3 Dioxin and Furan Matrix Spike Recovery (percent)

QAPP Table 2.3 specifies laboratory performance standard (LPC) for dioxin MS recovery as 70 percent. Only one recovery was less than 70 percent and that was for the congener OCDD which is not used in TEQ calculations and, therefore, the low recovery is inconsequential to the RG achievement decision. All other recoveries were greater than 80 percent. Median recoveries for all but one congener equaled or exceeded 100 percent. The one exception had a respectable median recovery of 96 percent. Overall the

MS recoveries were slightly higher than respective LCS recoveries suggesting a slight conservative high bias. The statistics are indicative of generally good measurement accuracy for all congeners.

Figure 2.4 presents the RPD for duplicate MS measurements. Note that the horizontal axis on this plot is a logarithmic scale. Table 2.3 of the QAPP specifies the laboratory performance standard (LPC) for dioxin MS RPD as 20 percent or less. Approximately 2.5 percent of RPDs exceed the 20 percent LPC and the median RPDs for all congeners are less than or equal to 7 percent. These statistics are very similar to the analogous statistics representing LCS and suggest that there is at most a minor effect of the matrix on measurement precision. Overall, the RPD measurement statistics indicates good analytical precision.

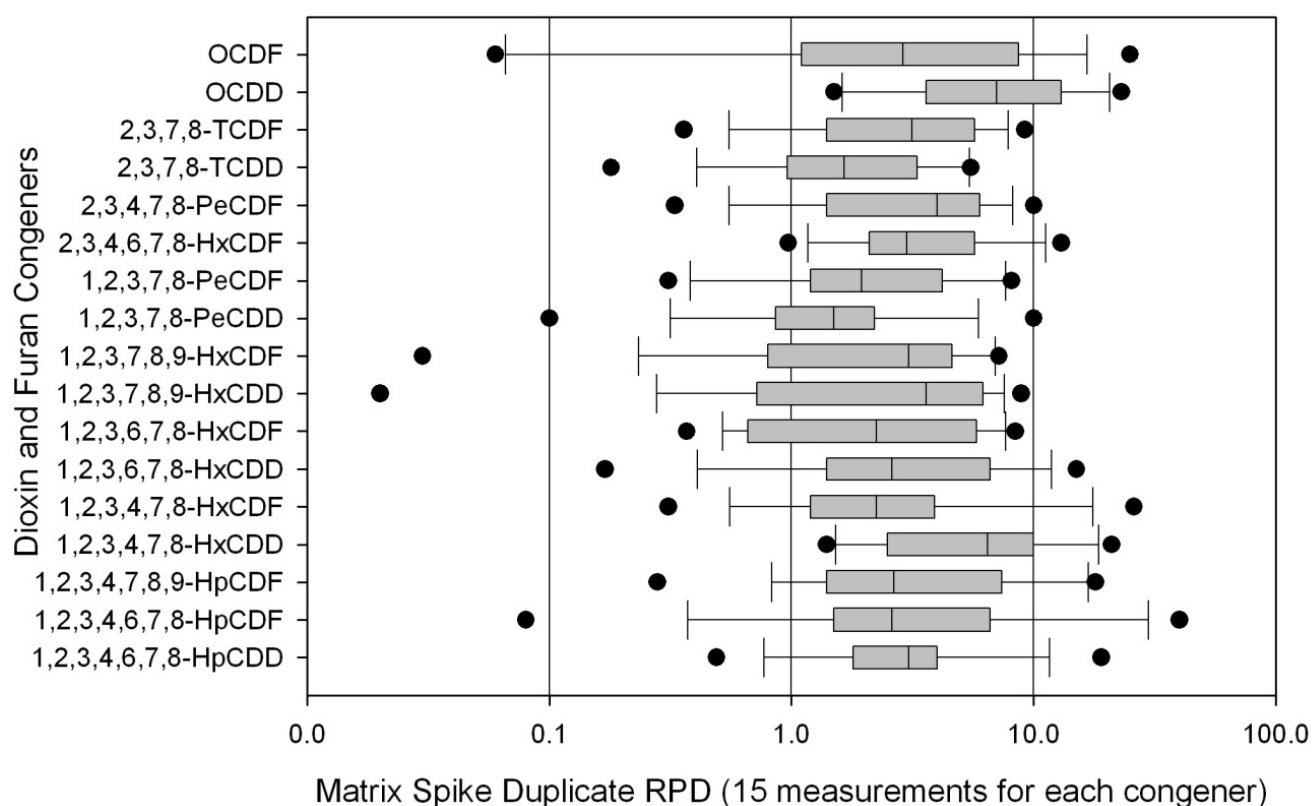


Figure 2.4 Dioxin and Furan Matrix Spike Duplicate RPDs

West Site Grid B03

MS and MSD test results for WS-B03-110825, presented in Table 2.2 exhibit recoveries between 56 and 120 percent and RPDs between 0 and 9 percent. The QAPP-specified laboratory control limits for recovery are 70 and 130 percent and RPD less than 35 percent. Antimony was the only analyte with an exceedance of a control limit. Note that spikes used to evaluate aluminum and magnesium were less

than 3 percent of the samples original concentration, thereby invalidating matrix spike analyses for these analytes.

The high antimony LCS recovery coupled with the low MS recovery suggest that the Site soil matrix may interfere with the measurement of antimony. This was observed in previous measurements of antimony which exhibit an average MS recovery of approximately 56 percent (DUR 2, Appendix A, Table SB-1). Tests performed by NPS in 2008 established that the recovery of antimony from background samples was approximately the same in 2008 as it was during the RI. There has not been a notable change in the sampling and analysis process since 2008, so it is expected that the antimony recovery for WS-B03-110825 is likewise approximately the same as typical RI recoveries and reflect a measurement accuracy that has remained unchanged throughout remediation. Because the antimony RG is a background-based value calculated using measurements made during the RI, and the comparability with CVS measurements has been established, the measurement interference is considered insignificant to the RG decisionmaking process. Discussion related to the comparability of inorganic parameter measurements during the RI to those made during the RAWP is presented in DUR 1, Section 4.8.

Laboratory Internal and Recovery Standards

Dioxins and Furans

An internal standard consisting of nine carbon-13 labeled (13C-labeled) congeners were added to all samples, blanks and QC samples before extraction and were used to quantitate the concentration of the analytes. Additionally, a recovery standard that consisted of two 13C-labeled congeners was introduced to each sample extract prior to injection into the high-resolution gas chromatograph and high resolution mass spectrometer measurement system and used to determine the percent recovery of the internal standards.

Internal and recovery standards were applied to all samples by the laboratory as part of its standard procedure for application of SW 846 Method 8290. The calculated recoveries were reviewed in accordance with the laboratory QAP and also during the data validation process and routinely found to be within acceptable limits.

West Site Grid B03

Initial calibration, continuing calibration, calibration blank standards, and ICP interference check samples were evaluated when testing WS-B03-110825, and the results are discussed in the associated laboratory report. No qualifiers indicating concern regarding the performance of these standards were assigned to WS-B03-110825 inorganic parameters data.

Laboratory Duplicates

Dioxin and Furans

The dioxin/furan CVS soil samples were sent from the Site to CT Laboratories in Baraboo, Wisconsin where they were ground, homogenized, and split according the FSP procedure prior to being mailed to the Test America Laboratory in Sacramento California for dioxin/furan analyses. Duplicate splits were created at CT Laboratories following the grinding and homogenization process. The results of analyses performed on these split samples are discussed in Section 2.6 with regard to field QC.

West Site Grid B03

Laboratory-generated splits of sample WS-B03-110825 were created following grinding and homogenization. These splits were analyzed for the various inorganic analytes in the parameter group, with calculated RPDs ranging from 0 to 15 percent for the various analytes (See Table 2.2). Mercury, selenium and cadmium were not detected in the sample or the duplicate. The QAPP establishes a maximum value of 20 percent for laboratory duplicate RPD. Past duplicate performance is presented in DUR 2, Section 2.4 and provides insight regarding overall metals analyses reproducibility. Suffice it to say that precision defined by past measurement is consistent with RPD measurements on WS-B03 splits. Intermittent precision errors were noted in previous DURs and likely persist because their cause was not discovered. Precision error is incorporated in the calculation of the reliance level and considered in the evaluation of WS-B03-110825 data usability presented in Section 2.8.

Laboratory QC Summary

Dioxin and Furans

The calculated holding times, recoveries and RPDs for LCS, LCSD, MS, MSD, KNO₃ and surrogate measurements suggest that the laboratory measurement of dioxins/furans are generally of good quality. The data validation reports, which reviewed internal laboratory QC steps and results, also concluded the laboratory measurements are of good quality. As needed, during data validation and subsequent data quality evaluations (DQEs 1, 2 and 3), data qualifiers were assigned to dioxin and furan data that demonstrated exceedance of LPCs. These qualifiers are not in the October 7, 2011 database but will be included in the final database.

West Site Grid B03

Laboratory performance was measured, in part, by evaluation of the LCS, LCSD, MS, MSD, and KNO₃ measurements and comparison of results to QAPP specified MQOs and LPCs. The results indicate that inorganic parameter concentration measurements made for the West Site Grid B03 CVS sample WS-B03-110835 has quality that is generally compliant with QAPP requirements.

2.6 Field QC

The discussion below details how measurements of precision and accuracy made using field QC exhibit MQO achievements that generally parallel LPC achievements. Data quality was routinely evaluated by including certified reference materials (CRMs) and splits of previously homogenized and tested samples in field prepared CVS batches. Concentrations in CRMs were blind to the testing laboratory. Measurements were not used to modify procedures or other operations related to the analysis of dioxin and furan or the WS-B03-110825 CVS, but rather provide a means to measure overall process performance.

The October 7, 2011 database contains the following new field QC results: 1) splits of laboratory homogenized CVS, and 2) EQIS-purchased certified reference materials (EQIS CRMs). Twenty-eight analyses on 14 duplicate sets of a CRM that contained 3 pg/g 2,3,7,8 TCDD were performed prior to the start of CVS testing and are pertinent to evaluation dioxin/furan data quality. The results of analyses performed on these samples are presented in the Krejci Database dated August 3, 2006 but are not included in the October 7, 2011 database. Results of analyses performed on these duplicate CRM sets are evaluated herein. Select QC data are tabularized and presented with statistical summaries in this section.

CRM Analyses

CRMs are commercially prepared and purchased soil samples containing a measured quantity of contaminant. CRMs containing dioxins were submitted to the laboratory with CVS and tested to evaluate measurement accuracy. Analyses of duplicate sets of CRMs provide data to calculate RPD for precision evaluation. Only NPS experts and EQIS knew the type and quantity of contaminants in the CRMs. The NPS's vendor-supplied certificates state the "made to" concentrations and the 95 percent acceptance limits for recovery for each parameter. The EQIS vendor supplied 99 percent acceptance limits for recovery of each included parameter. These 99 percent limits were used to back-calculate the 95 percent acceptance limits that are subsequently used for comparison with laboratory measurements. CRMs were submitted prior to the start of CVS (Initial CRM Testing) and periodically during the RAWP (Continued CRM Testing). A CRM was not submitted for analysis with sample WS-B03-110825 but many CRMs were previously submitted for metals analyses as discussed in DUR 2 and DUR 1. These previous analyses will be used with laboratory QC test results in Section 2.8 to assess data usability of the WS-B03-110825 inorganic analysis results.

Initial CRM Testing

Prior to the start of CVS testing, dioxin/furan analyses were performed at the Test America Sacramento laboratory on 14 duplicate sets of CRMs that had a "made to" concentration of the congener 2,3,7,8 TCDD equal to 3 pg/g. Table 2.3 presents the 28 test results.

Table 2.3 Results of Test on 14 Duplicate CRM Sets ("Made to" Concentration was 3 pg/g)

Laboratory Batch Identification Number	Date Received	Analysis Method	Date of Analysis	Chemical Name	Chemical Concentration	Concentration Unit
5329257	11/14/2005	SW8290	12/1/05	2,3,7,8 TCDD	4.4	pg/g
5336345	11/8/2005	SW8290	12/8/05	2,3,7,8 TCDD	3.1	pg/g
5336345	11/8/2005	SW8290	12/8/05	2,3,7,8 TCDD	2.2	pg/g
5336345	11/9/2005	SW8290	12/8/05	2,3,7,8 TCDD	2.9	pg/g
5336345	11/9/2005	SW8290	12/8/05	2,3,7,8 TCDD	2	pg/g
5336345	11/9/2005	SW8290	12/8/05	2,3,7,8 TCDD	2.8	pg/g
5336345	11/9/2005	SW8290	12/8/05	2,3,7,8 TCDD	2.3	pg/g
5336345	11/9/2005	SW8290	12/8/05	2,3,7,8 TCDD	2.3	pg/g
5336345	11/9/2005	SW8290	12/8/05	2,3,7,8 TCDD	2.1	pg/g
5336345	11/9/2005	SW8290	12/8/05	2,3,7,8 TCDD	2.1	pg/g
5336345	11/9/2005	SW8290	12/8/05	2,3,7,8 TCDD	2.3	pg/g
5336345	11/8/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.1	pg/g
5336345	11/8/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.6	pg/g
5336345	11/8/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.4	pg/g
5336345	11/8/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.9	pg/g
5336345	11/9/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.4	pg/g
5336346	11/10/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.5	pg/g
5336346	11/10/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.9	pg/g
5336346	11/10/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.4	pg/g
5336346	11/10/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.4	pg/g
5336346	11/10/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.4	pg/g
5336346	11/10/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.4	pg/g
5336346	11/10/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.8	pg/g
5336345	11/14/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.4	pg/g
5336345	11/14/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.7	pg/g
5336345	11/14/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.3	pg/g
5336345	11/14/2005	SW8290	12/9/05	2,3,7,8 TCDD	2.8	pg/g
5336346	11/10/2005	SW8290	12/10/0	2,3,7,8 TCDD	3.1	pg/g

These tests were performed prior to the start of testing for the purpose of evaluating laboratory initial performance. The mean and standard deviation of the measured concentrations are 2.57 pg/g and 0.47 pg/g, respectively. The data suggest that measurements are biased approximately 14 percent low ($[(3.0 - 2.57)/3.0] \times 100$). The calculated coefficient of variation (CV) is equal to approximately 18 percent ($[(0.47/2.57) \times 100]$). The CRM vendor specified acceptance range for measurements was 2.1 pg/g to 3.9 pg/g. Three measurements equal to 2.1 pg/g are at the lower extreme of this range and two measurements are outside the range, one high and the other low. Consequently, the MQO requiring routine measurements within CRM vendor's limits was not achieved. Repeated duplicate analyses from such a distribution is expected to exhibit RPDs less than 35 percent approximately 80 percent of the time. The QAPP-specified MQO for duplicates requires that the RPD routinely be less than 35 percent. Consequently, the expected 80 percent achievement rate does not satisfy this MQO.

Continued CRM Testing

In addition to the initial CRM tests described above, a CRM was submitted by EQIS at a frequency of one for every twenty samples collected resulting in the analysis of 12 CRMs. According to the QAPP, CRMs were to contain congeners at concentrations more than half and less than twice the RG so that accuracy and precision of measurements reflect the concentration range of concern. The first 11 CRMs submitted contained concentrations of congeners that were nearly four times the RG and the last CRM contained dioxin and furan congeners at concentrations approximately 200 times the RG. The exceedance of the QAPP maximum concentration requirement for the 11 CRMs is not expected to significantly impact the value of the CRM analyses for assessing measurement accuracy; however, the concentration of the last CRM was too excessive to result in a measurement that has precision and accuracy representative of characteristics near the RG, and, therefore, the last CRM is not included in the data set summarized and discussed below. Suffice it to say that all measured congener concentrations for the twelfth CRM were within vendor specified control limits. The results of tests on the first 11 CRMs are presented in Table 2.4. General statistics related to the data set are presented in Table 2.5.

Table 2.4 Result of Tests on Dioxin/Furan CRMs Submitted from the Field

Congener	CRM Certified Concentrations			Sample Identification										
	CRM "Assigned Gravimetric Value"	Upper Control Limit	Lower Control Limit	WS-Z34-100419A	WS-Z34-100419B	WS-Z35-100811	WS-Z36-100811	WS-Z39-100923	WS-Z39-100923 DUP	WS-Z40-101006	WS-Z41-101014	WS-Z42-101117	WS-Z44-110316	WS-Z45-110427
1,2,3,4,6,7,8-HpCDD	10.5	20.7	0.341	9.2	8.9	9.6	11	12	14	10	11	13	12	11
1,2,3,4,6,7,8-HpCDF	11.5	22.1	0.892	9.8	9.2	10	11	11	14	13	7.7	10	10	8.7
1,2,3,4,7,8,9-HpCDF	11.4	22	0.837	8.3	7.2	9.6	9.2	10	12	9.1	7.4	9.9	7.5	5.4
1,2,3,4,7,8-HxCDD	7.25	15.9	0	8.9	8.6	10	8.2	9.9	13	11	12	8.1	12	11
1,2,3,4,7,8-HxCDF				10	9.1	8.3	7.1	9.4	8.4	11	10	8	9.9	8.1
1,2,3,6,7,8-HxCDD	10.9	21.2	0.561	7.3	5.9	7.4	8.1	8.1	10	9.6	8.1	4.5	7.1	9.1
1,2,3,6,7,8-HxCDF	11.6	22.3	0.947	10	9.2	9.6	11	10	14	12	14	11	12	9.2
1,2,3,7,8,9-HxCDD	9.86	19.7	0	7.7	6.4	8.9	8.1	9.2	11	10	7.7	5.8	10	6.9
1,2,3,7,8,9-HxCDF	12.1	22.3	1.22	6.9	6.4	8.9	8.4	10	12	8.3	10	8.8	10	8.2
1,2,3,7,8-PeCDD	11.3	21.1	0.782	9.6	7.5	9.7	11	12	12	12	11	8.7	10	11
1,2,3,7,8-PeCDF	10.9	21.2	0.561	13	11	9.7	11	12	12	12	15	11	11	11
2,3,4,6,7,8-HxCDF	10.6	20.8	0.369	9.9	9	8.8	9.9	11	12	12	11	9.1	9.9	8.3
2,3,4,7,8-PeCDF	11	21.4	0.616	9.2	8.1	7.7	9.3	11	11	12	11	8.6	10	8.1
2,3,7,8-TCDD	11.5	22.1	0.892	9.2	7.9	9	9.6	11	9.1	11	10	8.8	12	10
2,3,7,8-TCDF	9.67	19.5	0	11	8.8	11	11	9.7	11	12	14	14	9.5	13
OCDD	11.3	21.8	0.782	20	16	20	30	74	44	27	24	38	24	22
OCDF	10.6	20.8	0.369	9.7	7.7	8.4	9	8	11	10	7.8	10	7.8	7.4

Table 2.5 Statistics Related to Tests on Dioxin/Furan CRMs Submitted from the Field

Congener	CRM "Assigned Gravimetric Value"	Mean	Percent Bias	Standard Deviation	Coefficient of variation
1,2,3,4,6,7,8-HpCDD	10.5	11.1	5.4%	1.6	14.5%
1,2,3,4,6,7,8-HpCDF	11.5	10.4	-9.6%	1.8	17.4%
1,2,3,4,7,8,9-HpCDF	11.4	8.7	-23.8%	1.8	20.5%
1,2,3,4,7,8-HxCDD	7.25	10.2	41.3%	1.7	16.4%
1,2,3,4,7,8-HxCDF		9.0		1.2	12.7%
1,2,3,6,7,8-HxCDD	10.9	7.7	-28.9%	1.6	20.6%
1,2,3,6,7,8-HxCDF	11.6	11.1	-4.4%	1.7	15.7%
1,2,3,7,8,9-HxCDD	9.86	8.3	-15.5%	1.6	19.6%
1,2,3,7,8,9-HxCDF	12.1	8.9	-26.4%	1.6	17.6%
1,2,3,7,8-PeCDD	11.3	10.4	-7.9%	1.5	14.0%
1,2,3,7,8-PeCDF	10.9	11.7	7.3%	1.4	11.9%
2,3,4,6,7,8-HxCDF	10.6	10.1	-4.9%	1.3	12.6%
2,3,4,7,8-PeCDF	11	9.6	-12.4%	1.5	15.1%
2,3,7,8-TCDD	11.5	9.8	-14.9%	1.2	12.1%
2,3,7,8-TCDF	9.67	11.4	17.5%	1.7	15.3%
OCDD	11.3		172.7%	16.5	
OCDF	10.6	8.8	-17.0%	1.2	13.7%

Values outside the CRM vendor's acceptance limits are indicated by bold font in Table 2.4. Note that OCDD measurements were routinely biased high and all other measurements were within the vendor's acceptance limits. The high bias for OCDD is inconsequential to data usability because OCDD was not used to develop the RG and is not used to calculate the TEQ for comparison to the RG. Due to its unique behavior and its insignificance to decisionmaking, discussion in the next two paragraphs related to the CRM data set excludes consideration of OCDD measurements.

Calculated values of standard deviation for individual congeners are presented in Table 2.5 and ranged between 1 and 2 pg/g. However, 11 is an insufficient number of measurements to obtain a good estimate of standard deviation. Therefore, the individual estimates were pooled to calculate a standard deviation of 1.53, which yields a CV of approximately 16 percent. This is very similar to an 18 percent CV calculated for the 2,3,7,8 TCDD congener using the previously discussed 28 initial CRM measurements. This suggests that analytical precision likely did not change significantly during CVS sampling and testing.

Observe on Table 2.5 that bias for different congeners ranged from approximately 48 percent high to about 29 percent low. For the congener 2,3,7,8 TCDD, the calculated bias is approximately 15 percent low. This is very similar to the value of 14 percent calculated at the start of testing using 28 CRM measurements, thereby, suggesting that dioxin/furan measurement accuracy likely did not change significantly during the RA. However, eleven is an insufficient number of measurements to obtain a good estimate of the mean; therefore, an improved estimate of bias is calculated by dividing the average of measurements for all congeners by the average of the CRM vendors assigned value. Such analysis presumes each congener inherently has approximately the same measurement accuracy, which may or may not be the case. Notwithstanding the possible error caused by this assumption, the calculated bias is approximately nine percent and low. This is a reasonably small bias and suggests that the dioxin/furan congener measurements are reasonably accurate.

Duplicate Analyses

Duplicate analyses were performed on splits of CVS samples. As a general routine, CT Laboratory homogenized and split samples and then returned three splits of each CVS sample to the Field Quality Assurance Officer (FQAO) who, in turn gave two of those splits to the NPS OSR. For all analytical methods except those for dioxin/furan analysis, the FQAO and NPS OSR each independently returned splits blind to the laboratory for analysis at a frequency of approximately one for every twenty CVS samples. The routine was slightly different for dioxin/furan analyses. For dioxin/furan analyses, duplicate splits of samples were created at CT Laboratories in Baraboo, Wisconsin, at the time when samples were ground and homogenized. These were then mailed with parent samples to Test America laboratory in Sacramento, California for testing. Each duplicate set underwent the same digestion and analysis processes as the original sample split. Because the duplicates were submitted for testing at the same time as their parent sample, they were tested in the same analytical batch as the parent material. Results of dioxin duplicate measurements and calculated RPDs are presented in Tables 2.6 and 2.7, respectively.

Italicized entries in Table 2.6 indicate that the congener was not detected and the value presented is the reporting limit. The RPDs calculated and presented in Table 2.7 represent conditions in which one or both measurements were above the reporting limit. When the value for one congener was less than the reporting limit, its reporting limit was used for the RPD calculation, thereby biasing the calculated RPD towards a low value.

High RPDs associated with 1,2,3,4,6,7,8 HpCDD, OCDD and 2,3,7,8 TCDF are inconsequential to decisionmaking because these congeners are not used to calculate the TEQ. High RPDs for other congeners exceed the QAPP-specified MQO limit of 35 percent; however, CVS results demonstrating high RPDs were seldom associated with CVS batches containing samples ultimately used for the RG achievement decision. Also, the congeners associated with high RPDs never accounted for more than 10.5 percent of the calculated TEQ in samples ultimately used for the RG achievement decision. Consequently, the observed RPD exceedances of the MQO are considered a minor quality concern.

A statistical analysis was performed on the CVS results for the dioxin/furan congener 2,3,7,8 TCDD; 2,3,7,8 TCDD is the most significant congener in determining RG achievement, because it has highest TEQ weight (i.e., the highest toxicity), and it was the most persistent congener measured on Site. The standard deviation of the 2,3,7,8 TCDD measurements calculated by pooling the duplicate information is 0.566 pg/g, and the mean is calculated to be 2.59 pg/g. These values are expectedly slightly larger than values calculated for the initial CRMs which had a standard deviation of 0.473 pg/g and a mean of 2.57 pg/g. A slightly higher variance for duplicates than for CRM results is expected, because CRMs are meticulously manufactured samples of known concentration created using easily homogenized cohesionless soil. In contrast, Site soils have unknown and potentially highly variable concentrations and consist of a clay soil that requires greater effort to completely homogenize.

2.7 Data Qualifiers

Data qualifiers are alphanumeric notations assigned to a piece of data to indicate a specific concern with that data. A general discussion of the Krejci Site process of data validation, verification, and qualification is provided in DUR 1, Section 7.6 and is incorporated herein by reference. CVS and QC measurements entered into the October 7, 2011 database after December 16, 2010 were evaluated in accordance with these validation, verification, and qualification procedures in DQE 3.

Dioxin and Furans

Dioxin/furan analyses achieved all LPCs and MQOs except that the desired precision as measured by field duplicates and CRMs were not routinely achieved. It is of considerable significance that this qualification is, in most cases, caused by RPD measurements exceeding 35 percent for congeners other than 2,3,7,8 TCDD. This is important because 2,3,7,8 TCDD is the primary contributor to the calculated TEQ and in most cases was the only congener detected in samples ultimately used for the RG achievement decision. Consequently, concerns regarding imprecision are diminished by observing that congener 2,3,7,8 TCDD exceedances of the precision MQOs are small and infrequent.

West Site Grid B03

WS-B03-110825 inorganic analyses were qualified for some analytes, as indicated in DQE 3. Section 2.8 evaluates the usability of these measurements using a statistical approach, and Section 3 summarizes the results of the data usability evaluation.

2.8 Reliance Level

The reliance level (RL) is a calculated concentration, unique to each analyte, used to determine if the measurement is sufficiently close to the RG to warrant closer examination of the measurement quality. In the case of dioxins and furans, the RL is unique to the calculated TEQ. The RL sets a limit on how close a CVS measurement can be to the RG without undue concern that the true sample concentration exceeds the RG excessively. A discussion about the use of RLs, as well as the derivation of RLs for this project, is provided in DUR 1, Section 7 and is incorporated herein by reference. Suffice it to say here that qualified measurements below the RL (and by definition below the RG) may be used with confidence to conclude the RG has been attained, and qualified measurements above the RL may be used with confidence to conclude the RG has not been attained. For qualified measurements between the RL and RG, additional evaluation is needed to determine with confidence that the measurement has sufficient quality to be used to establish that RG achievement.

Dioxins and Furans

An RL was calculated to aid in the evaluation of the usability of dioxin/furan test results for RG achievement decisions. The calculation was initially presented in DUR 1, Section 8.4, and was based solely on estimates of precision and accuracy representing initial CRM test results reported in the September 11, 2009 database. The RL was calculated in terms of the TEQ to be 2.7 pg/g.

Results of CRM and duplicate sample analyses that were performed after completion of the initial excavation provide for improved estimates of dioxin/furan measurement precision and accuracy. These are used as follows to calculate a new RL. Because 2,3,7,8 TCDD is the predominant contributor to the calculated TEQ for samples ultimately used for the RG achievement decision, the statistics selected for RL calculation are those determined specifically for the 2,3,7,8 TCDD congener.

The RL calculation requires estimates of precision and accuracy. A pooled estimate of standard deviation calculated using both the 12 CVS duplicate RPDs and the 28 initial CRM test results is calculated to be 0.50 pg/g. Standard deviations calculated independently for the two data sets are presented in Section 2.6. The average recoveries of the 2,3,7,8 TCDD congener from the 28 initial CRMs and 11 subsequent CRMs are calculated to be 86.4 percent and 85.2 percent respectively. A pooled estimate of the recovery is 86.1 percent and this is subsequently used to represent accuracy for RL calculation.

An RL equal to 2.7 pg/g is calculated according to methodology presented in DUR 1 using a value of 0.50 to represent the standard deviation and a value of 86.1 percent to represent the expected recovery. This is the same as the value 2.7 pg/g initially calculated in DUR 1 and this consistency provides added

confidence that the DUR 1 assessment of dioxin/furan data usability was good. Such confidence is important, because DUR 1 data indicated that some dioxin/furan remediation areas achieved the RG and, consequently, no subsequent dioxin/furan remediation was performed in those areas.

Table 2.6 Results of Dioxin/Furan Measurements for Duplicate Splits

	ES-A01		WS-B04		WS-H02		WS-G02		WS-D03		WS-F07		WS-J03		WS-D03		WS-H05		WS-G04		WS-I02		WS-D04		WS-D04	
	DUP 21	ES-A01-080623	DUP 20	WS-B04-080626	DUP-49	WS-H02-100407	DUP 49	WS-H02-100802	DUP 50	WS-D03-100803	DUP-52	WS-F07-100923	DUP-54	WS-I03-101006	DUP-55	WS-D03-101013	DUP-56	WS-H05-101007	DUP-58	WS-G04-110317	DUP-59	WS-I02-110504	DUP-60	WS-D04-110607	DUP-61	WS-D04-110712
Congener																										
1,2,3,4,6,7,8-HpCDD	110	110	190	220	6.2	6.5	17	7.1	20	23	64	49	11	11	2.7	4.2	12	11	0.31	0.32	0.155	0.145	0.38	0.25	0.145	0.17
1,2,3,4,6,7,8-HpCDF	18	19	8.7	8.2	0.34	0.55	0.7	0.42	0.6	0.65	5.7	2.7	0.405	0.435	0.18	1.6	0.285	0.205	0.019	0.02	0.028	0.06	0.125	0.048	0.023	0.036
1,2,3,4,7,8,9-HpCDF	0.41	0.5	0.65	0.6	0.095	0.14	0.07	0.033	0.16	0.115	0.205	0.15	0.06	0.06	0.09	0.28	0.06	0.065	0.011	0.015	0.035	0.038	0.07	0.06	0.027	0.023
1,2,3,4,7,8-HxCDD	0.405	0.47	0.55	0.6	0.055	0.08	0.085	0.09	0.19	0.24	0.24	0.26	0.19	0.065	0.045	0.09	0.055	0.06	0.026	0.05	0.038	0.038	0.065	0.065	0.025	0.026
1,2,3,4,7,8-HxCDF	2.5	0.65	3.1	2.6	0.23	0.245	0.35	0.105	0.285	0.38	0.55	0.41	0.325	0.205	0.155	0.255	0.205	0.185	0.014	0.015	0.034	0.055	0.055	0.041	0.014	0.014
1,2,3,6,7,8-HxCDD	4.6	6.4	2	2	0.12	0.145	0.185	0.15	0.55	0.7	0.45	0.355	0.17	0.155	0.23	0.21	0.22	0.195	0.046	0.043	0.033	0.03	0.1	0.06	0.037	0.029
1,2,3,6,7,8-HxCDF	0.6	0.55	0.41	0.32	0.09	0.125	0.12	0.085	0.155	0.28	0.405	0.33	0.12	0.125	0.07	0.15	0.08	0.026	0.003	0.012	0.025	0.039	0.04	0.03	0.013	0.013
1,2,3,7,8,9-HxCDD	4	4.3	3.8	4.2	0.46	0.48	0.65	0.6	0.55	0.7	3.7	3.4	3.5	2.9	0.275	0.325	6.8	6.3	0.14	0.14	0.08	0.065	0.155	0.135	0.065	0.085
1,2,3,7,8,9-HxCDF	0.07	0.09	0.065	0.095	0.028	0.05	0.016	0.012	0.016	0.022	0.022	0.042	0.045	0.01	0.055	0.055	0.034	0.046	0.004	0.016	0.036	0.055	0.06	0.044	0.015	0.019
1,2,3,7,8-PeCDD	0.44	0.5	0.415	0.405	0.235	0.26	0.26	0.24	0.35	0.43	0.3	0.285	0.205	0.25	0.275	0.225	0.275	0.305	0.245	0.225	0.125	0.09	0.17	0.215	0.075	0.085
1,2,3,7,8-PeCDF	0.38	0.315	0.185	0.155	0.16	0.16	0.15	0.11	0.115	0.14	0.4	0.35	0.15	0.185	0.06	0.075	0.055	0.065	0.016	0.033	0.06	0.085	0.065	0.06	0.03	0.026
2,3,4,6,7,8-HxCDF	0.495	0.465	0.305	0.23	0.115	0.165	0.175	0.125	0.145	0.185	0.34	0.3	0.135	0.11	0.095	0.105	0.075	0.09	0.004	0.013	0.029	0.045	0.046	0.035	0.013	0.014
2,3,4,7,8-PeCDF	0.6	0.55	0.305	0.23	0.155	0.185	0.225	0.16	0.185	0.31	0.5	0.55	0.17	0.25	0.095	0.08	0.105	0.07	0.017	0.033	0.06	0.085	0.065	0.06	0.033	0.029
2,3,7,8-TCDD	0.66	0.7	2.8	2.7	3.5	3.4	2.5	2.6	3.9	3.8	1.2	1.2	2	2.4	3.1	3.2	2.5	2.2	5.6	5.3	1.9	2	3.2	3.2	1.4	1.5
2,3,7,8-TCDF	1.5	1.5	0.37	0.33	0.115	0.43	0.4	0.055	0.031	0.41	1.7	1.7	0.46	0.53	0.155	0.4	0.5	0.49	0.042	0.048	0.07	0.1	0.18	0.18	0.018	0.019
OCDD	20000	26000	50000	50000	710	760	3100	800	2000	2400	9700	7700	1000	1000	170	170	1500	1500	56	53	8.8	8.4	19	15	6.3	6.4
OCDF	17	20	31	38	0.305	0.48	0.415	0.25	1.45	0.95	4.5	1.3	0.275	0.35	0.175	14	0.29	0.225	0.033	0.055	0.065	0.165	0.185	0.085	0.05	0.08
TEQ	2.67	2.75	4.32	4.16	3.82	3.77	2.92	2.93	4.37	4.44	2.25	2.17	2.65	3.02	3.38	3.51	3.44	3.08	5.76	5.46	2.02	2.13	3.37	3.38	1.47	1.58
RPD		3.0%		3.8%		1.3%		0.4%		1.5%		3.6%		13.2%		3.6%		11.0%		5.3%		4.9%		0.2%		6.8%

Table 2.7 RPD's Calculated for Duplicate Splits

Congener	DUP 21	DUP 20	DUP-49	DUP 49	DUP 50	DUP-52	DUP-54	DUP-55	DUP-56	DUP-58	DUP-59	DUP-60	DUP-61
1,2,3,4,6,7,8-HpCDD	0	15	5	82	14	27	0	43	9				
1,2,3,4,6,7,8-HpCDF	5	6				71		160					
1,2,3,4,7,8,9-HpCDF													
1,2,3,4,7,8-HxCDD													
1,2,3,4,7,8-HxCDF	117	18											
1,2,3,6,7,8-HxCDD	33	0											
1,2,3,6,7,8-HxCDF													
1,2,3,7,8,9-HxCDD	7	10				8	19		8				
1,2,3,7,8,9-HxCDF													
1,2,3,7,8-PeCDD													
1,2,3,7,8-PeCDF													
2,3,4,6,7,8-HxCDF													
2,3,4,7,8-PeCDF													
2,3,7,8-TCDD	6	4	3	4	3	0	18	3	13	6	5	0	7
2,3,7,8-TCDF	0	11	116	152		0	14	88	2				
OCDD	26	0	7	118	18	23	0	0	0	6	5	24	2
OCDF	16	20				110							

West Site Grid B03

Derived RLs for inorganic parameter analyses were initially calculated and presented in DUR 1, Appendix A. Updated values were calculated and presented in DUR 2, Appendix A using QC associated with all CVS, except data presented herein for WS-B03-110825. The RL values presented in the two previous DURs were not significantly different and are presented in Table 2.8 along with RGs and WS-B03-110825 results.

The limited QC acquired with analysis of WS-B03-110825 does not result in a significant change to the DUR 2-derived RL, and QC discussed in Sections 2.5 and 2.6 indicate that quality associated with WS-B03-110825 is similar to that acquired for previous project analyses. Therefore comparison of the WS-B03-110825 analyte results to the DUR 2-derived RL is appropriate. As shown in Table 2.8, all WS-B03-110825 analyte results are below their respective RLs and, consequently, usable for determining RG achievement.

Table 2.8 Inorganic Analyte RGs, RL's, and WS-B05-110825 Results

Analyte	Tier 1 RG (mg/kg)	Tier 2 RG (mg/kg)	DUR 1 Reliance Level (mg/kg)	DUR 2 Reliance Level (mg/kg)	WS-B03-110825
ALUMINUM	21000	24000	19150	19453	14100
ANTIMONY	1.9		1.6	1.7	0.27
ARSENIC	13	30	34	34	13.8
BARIUM	210	220	235	233	88.3
BERYLLIUM	2.1		1.2	1.2	0.75
BORON	31	35	32.9	30	22.4
CADMIUM	0.57	1.3	0.59	0.56	0.012
CHROMIUM	31	35	29.6	30.4	18.1
COBALT	21	30	29	29	14
COPPER	34		34	34	23.2
LEAD	100		108.7	108.3	9.7
MANGANESE	3650		3987	3978	480
MOLYBDENUM	14	16	16.9	16.9	5.4
NICKEL	190		192	192	33.6
SELENIUM	1.9	14	10.2	10.2	0.12
SILVER	17	17	16.7	16.6	0.11
VANADIUM	37	44	32.5	33.4	6.1
ZINC*	140		139	139	73.1
MERCURY	1.7	2.4	2.62	2.62	0.02

* Two RLs were calculated for Zinc in DUR1 to isolate data quality problems associated with a single batch.

Section 3. Data Usability Summary

3.1 Dioxin and Furans

The dioxin/furan data is qualified because laboratory performance criteria and MQOs were occasionally not achieved. As explained previously, additional evaluation of qualified data is necessary to determine whether the data quality is sufficiently reliable to be used to determine RG achievement. One part of this evaluation is a statistical analysis conducted by comparing each qualified measurement to a derived

RL unique to each analyte. The other part of this evaluation includes a review of the manner in which the data was generated, assessment of various quality control measures, review of laboratory and data validation reports, DQE 3, field and lab audits, and the database itself.

Select quality control measurements and calculated RLs are presented in Section 2. The derived RL is 2.7 pg/g, and the RG is 3.0 pg/g. Appendix A presents the results of all dioxin/furan analyses for all grids, and compares the last dioxin/furan CVS TEQ result in each grid to the RLs and RGs. There are 7 individual ¼-acre grids one 1-acre dioxin/furan sampling area that have a CVS TEQ result that is less than or equal to the RG and greater than the RL. These results require additional evaluation to determine usability, and are listed in Table 3.1, below.

Table 3.1 Grids and Dioxin Sampling Areas having Calculated TEQs Less than the RG but Greater than the RL

West Site Grid or Sampling Area	TEQ (pg/g)	Laboratory Test Batch Number	2,3,7,8 TCDD Concentration (pg/g)
G6,G7,H6, I6	2.9	118261	2.3
F3	2.8	1131318	2.7
G2	2.9	237391	2.5
H2	2.9	237391	2.4
G4	2.8	1131318	2.6
H4	3	298279	2.8
J3	3	298279	2.4
J5	3	298279	2.4

It is important to note that of the 14 congeners used to calculate TEQ, only three congeners were detected in the last samples collected representing the grids listed in Table 3.1. Only 2,3,7,8 TCDD was detected in ¼-acre grids F3, G2, H2, G4 and H4. 2,3,7,8 TCDD represented more than 99, 89, and 90 percent of the detectable congeners in ¼-acre areas J5 and J3 and 1-acre area G6, G7, H6, and I6, respectively. Because 2,3,7,8 TCDD is overwhelmingly the primary contributor to the calculated TEQs, it is both necessary and sufficient to limit subsequent data usability evaluation to the 2,3,7,8 TCDD congener.

It is likewise important to recognize that for congeners not detected, half the detection limit was used in the TEQ calculation. The magnitudes of calculated TEQs varied as much as 21 percent due solely to variation between samples in detection limits. Generally, as the detection limits decreased the calculated TEQ values approached the 2,3,7,8 TCDD concentrations, which with one marginal exception are equal to or below the RL. This uncertainty in TEQ calculation caused by variation in measurement sensitivity is not considered in the calculation of RLs. Considering this, the significance of the difference between the RG and the RL is favorably diminished.

The samples used to calculate the nine TEQs listed in Table 3.1 were tested in four independent laboratory batches (batch numbers 118261, 1131318, 237391 and 298279). Therefore, evaluation of 2,3,7,8 TCDD measurements is focused on these specific batches.

Batch 118261:

WS-I06-100406 was collected to represent the approximately 1-acre dioxin/furan sampling area comprised of West Site grids G6, G7, H6 and I6. This sample was extracted and analyzed within QAPP-specified holding times. The batch in which this sample was tested included two MS/MSD samples. The calculated MS/MSD RPDs were 0.78 and 4.6 percent and the recoveries were 109, 112, 115 and 121 percent. The calculated LCS/LCSD RPD was 1.3 percent and the associated recoveries were 113 and 115 percent. The MS/MSD and LCS/LCSD data favorably suggests a conservative bias toward high 2,3,7,8 TCDD measurement with little or no matrix interference. A RPD of 16 percent was calculated for field duplicate CRMs. The 2,3,7,8 TCDD recovery for the two CRMs tested were 80 percent and 69 percent. The 69 percent value is the lowest CRM recovery measured and the 80 percent value is slightly lower than average. Although recovery is low, it is within manufacturer recommended acceptance limit and, therefore, achieves the respective MQO. The internal standard recovery for 13C-2,3,7,8 TCDD was 92 percent, and the recovery of all early eluting internal standards was greater than 83 percent. Laboratory and field quality control measures associated with measuring 2,3,7,8 TCDD in WS-I06-100406 demonstrate a high level of precision, suggesting that observations of imprecision that caused TEQ qualification are intermittent and likely do not adversely affect the test result. Consequently, the dioxin/furan measurements for WS-I06-100406 are considered acceptable for determining RG achievement.

Batch 1131318:

WS-F03-110427 and WS-G04-110426 were collected to represent the West Site dioxin/furan sampling grids F3 and G4, respectively. Both samples were extracted and analyzed within QAPP-specified holding times. The batch in which these samples were tested included one MS/MSD sample set, one LCS/LCSD data set, and a duplicate analysis of a CRM. The calculated MS/MSD RPD was 1.8 percent and the recoveries were 104 and 98 percent. The calculated LCS/LCSD RPD was 2.4 percent and the associated recoveries were 100 and 98 percent. The CRM was analyzed in duplicate yielding an RPD of 5.4 percent and 2,3,7,8 TCDD recoveries of 86 percent and 87 percent. CRM recovery is low but within manufacturer recommended acceptance limit and, therefore, achieves the respective MQO. A field duplicate yielded an RPD of 10 percent. The MS/MSD, LCS/LCSD, and duplicate data suggests little to no 2,3,7,8 TCDD measurement bias and reasonably good precision.

The grid G4 sample internal standard recovery for 13C-2,3,7,8 TCDD was 84 percent, and the recovery of all early eluting internal standards were 80 to 86 percent. The grid F3 sample internal standard recovery for 13C-2,3,7,8 TCDD was 66 percent and the recovery of all early eluting internal standards were 66 to 69 percent. 2,3,7,8 TCDD concentration measurements within the batch attained all LPC and MQOs for WS-F03-110427 and WS-G04-110426. The precision and accuracy measurements associated specifically with this batch indicate a high level of precision and accuracy. It is concluded that measurement

imprecision was intermittent and not likely to influence the 2,3,7,8 TCDD results for WS-F03-110427 or WS-G04-110426. Consequently, the dioxin/furan measurements for these samples are considered acceptable for determining RG achievement.

Batch 237391:

WS-G02-100802 and WS-H02-100802 were collected to represent the West Site dioxin/furan sampling grids G2 and H2, respectively. Both samples were extracted and analyzed within QAPP-specified holding times. High calculated TEQs for WS-G02-100802 and WS-H02-100802 were caused in part by detection limits for undetected congeners being substantially higher than average. When a congener is not detected it is presumed to be present at half the detection limit for TEQ calculation. The grid H2 and grid G2 TEQs are respectively 4 percent and 8 percent higher than would have been calculated if the average detection limits for the PCDD and PCDF congeners had been realized and the congeners remained undetected. Since grid H2 and grid G2 calculated TEQs are less than 7 percent higher than the RL, it is likely that a more sensitive analysis would, by itself, have resulted in the grid G2 sample TEQ being less than the RL. It is, therefore, reasonable to conclude that the grid G2 sample test results are likely of acceptable quality and the grid H2 test results are likely a better quality than the magnitude of RL exceedance suggests.

Batch 237391 included one MS/MSD sample set, one LCS/LCSD data set, two field duplicate sample sets, and a duplicate analysis of a CRM. The calculated MS/MSD RPD was 3.3 percent and the recoveries were 101 and 99 percent. The calculated LCS/LCSD RPD was 14 percent and the associated recoveries were 100 and 87 percent. The CRM was analyzed in duplicate, yielding an RPD of 12.9 percent and 2,3,7,8 TCDD recoveries of 78 percent and 84 percent. CRM recovery is low but within the manufacturer recommended acceptance limit and, therefore, achieves the respective MQO. The field duplicates yielded an RPD of 10 percent. The MS/MSD, LCS/LCSD, and duplicate data suggests little to no 2,3,7,8 TCDD measurement bias and reasonably good precision. The grid G4 sample internal standard recovery for 13C-2,3,7,8 TCDD was 107 percent, and the recovery of all early eluting internal standards were 102 to 118 percent. The grid H2 sample internal standard recovery for 13C-2,3,7,8 TCDD was 88 percent and the recovery of all early eluting internal standards were 88 to 96 percent. Batch 237391, in which WS-G02-100802 and WS-H02-100802 were tested, attained all LPC and MQOs related to 2,3,7,8 TCDD concentration measurements. It is concluded that the measurement imprecision was intermittent and not likely to influence the 2,3,7,8 TCDD results for WS-G02-100802 and WS-H02-100802. Consequently, the dioxin/furan measurements for these samples are considered acceptable for determining RG achievement.

Batch 298279:

WS-H04-101007, WS-J03-101006 and WS-J05-101006 were collected to represent the West Site dioxin/furan sampling grids H4, J03 and J05. All samples were extracted and analyzed within QAPP-specified holding times. TEQs for WS-J3-101006 and WS-J5-101006 were elevated, in part, by detection limits for undetected congeners being substantially higher than average. The grid H2 and grid G2 TEQs

are both approximately 15 percent higher than would have been calculated if the average detection limits for the PCDD and PCDF congeners had been realized and the congeners remained undetected.

The batch in which WS-H04-101007, WS-J03-101006 and WS-J05-101006 were tested included two field duplicate sample sets. One of these field duplicates, DUP 54, was a split of WS-J03-101006, and like the original sample resulted in a calculated TEQ that achieves the RG. Two measurements of WS-J03-101006 that achieve the RG provide confidence that the measurements for this sample are sufficiently accurate and precise to be used for RG determination. The RPD for the two duplicate sets were 3 and 18 percent.

Batch 298279 included one MS/MSD sample set, one LCS/LCSD data set, and a duplicate analysis of a CRM. The calculated MS/MSD RPD was 5.4 percent and the recoveries were 91 and 93 percent. The calculated LCS/LCSD RPD was 6.1 percent and the associated recoveries were 103 and 97 percent. The CRM was analyzed in duplicate yielding an RPD of 28 percent and 2,3,7,8 TCDD recoveries of 96 percent and 72 percent. One of the two CRM recoveries is low but within the manufacturer recommended acceptance limit and, therefore, achieves the respective MQO. The RPD for the two duplicate sets is desirably low, with values of 4 and 13 percent. The MS/MSD, LCS/LCSD, and duplicate data suggests a small, low 2,3,7,8 TCDD measurement bias and reasonably good precision. Cumulatively, this information leads to the conclusion that the 2,3,7,8 TCDD measurements representing WS-H04-101007, WS-J03-101006, and WS-J05-101006 have acceptable quality for making the RG achievement decision.

3.2 West Site Grid B03

A comparison of WS-B03-110825 analyte concentrations to DURs 1 and 2 RLs is provided in Table 2.8. All WS-B03-110825 analyte concentrations are below their respective RLs and consequently usable for determining RG achievement.

Section 4. RG Achievement

The CD Statement of Work (SOW) provides the following with regard to RG achievement:

Post-excavation characterization will be performed to verify that remaining soils meet the Remediation Goals (RGs) set forth in Appendix D. . . . One composite sample comprising 40 specimens collected on a grid pattern within each 1/4-acre will be analyzed for all parameters shown in Appendix D that are associated with the area, except for dioxin/furan (which is described in Step 7 below) and benzene. . . . The following verification criteria shall apply. For parameters with Tier-2 RGs, up to two exceedances of Tier-1 RGs are permitted for each 1/4-acre area, so long as the Tier-2 RGs are achieved for those parameters. For parameters without defined Tier-2 RGs, exceedance of a Tier-1 RG within any 1/4-acre area constitutes failure. In the event that a 1/4-acre verification sample fails either of these verification criteria, Ford may collect a resample from a multi-point grid with similar point spacing used in the original sample, offset to a new origin. The resample

would be analyzed for each parameter group that had an exceedance. For example, if there is an exceedance for one metal parameter, all metals would be analyzed on the resample. The parameter groups are: (1) metals; (2) volatile organic compounds; (3) polycyclic aromatic hydrocarbons; (4) pesticides and PCBs; (5) phthalate esters; and (6) dioxin/furan. The relevant parameter group for each parameter is shown in Appendix D. If the resample results satisfy the verification criteria for all parameters in the parameter group, the 1/4-acre area will be deemed to have achieved the RGs for all contaminants except dioxin/furan.

DURs 1 and 2 established that all grids other than West Site grid B3 have attained all RGs, except for grids with dioxin/furan results that were not yet evaluated. DUR 1 also established that West Site grid B3 achieved all RGs other than those for the inorganic parameter group. West Site grid B3 was subsequently excavated, and CVS sample WS-B03-110825 was collected and tested for analytes in the inorganic parameter group. Data quality analyses of the CVS results for sample WS-B03-110825 are presented in Sections 2 and 3 and support the conclusion that the results have acceptable quality and may be used to determine RG achievement. The CVS results demonstrate that West Site grid B3 has achieved all inorganic analyte RGs.

The SOW provision concerning achievement of the dioxin/furan RG (referred to as Step 7 in the previous SOW citation) includes the following, in relevant part:

Dioxin/furan sampling and analysis will be conducted as follows, unless Ford and the NPS mutually agree to a different methodology. Verification sampling for the dioxin/furan RG will be conducted in areas R1, R2, and R3 only, after RGs have been achieved in these areas for the other 38 parameters or it has been determined that no further excavation for those parameters is required. . . . A 4-part unbiased composite sample will be collected from 1-acre sections of these areas. . . . If initial dioxin/furan sampling results reveal an exceedance of the dioxin/furan RG, Ford may resample the area by collecting a 40-specimen composite from each ¼-acre area in the original acre. If the resample results satisfy the RG for dioxin/furan, the ¼-acre section will be deemed to have achieved that RG, and no further excavation will be required. . . .

As indicated in the above quotation by reference to “areas R1, R2, and R3,” there are twelve (12), one-acre dioxin/furan test areas on the Site. See DUR 1, Figures 2.2 and 2.3. As detailed in the SOW, initially each 1-acre area (rather than its four component ¼-acre grids) was represented by a single, multi-increment dioxin/furan sample. The SOW provided that dioxin/furan sampling would not be conducted until after RGs had been achieved in the tested area for all other parameters, but NPS did not object when EQIS requested the opportunity to conduct earlier dioxin/furan sampling, because the provision was included for efficiency purposes and early dioxin/furan sampling would cause no harm. Most of the 1-acre grids initially failed to achieve the dioxin/furan RG and were subsequently sampled by their respective ¼-acre grids, as provided in the SOW.

Appendix A compares the dioxin/furan RG (3.0 pg/g 2,3,7,8 TCDD TEQ) to the CVS results (as calculated TEQs) contained in the October 7, 2011 database. Dioxin/furan CVS results that were evaluated in DQE 1 are presented in Appendix A along with all subsequent dioxin/furan CVS results. The data is presented for each congener and each ¼-acre grid or 1-acre dioxin/furan sampling area, as applicable. Column labels are sample identifiers, and each column presents results from a separate sampling event for the subject grid or area. Sample identifiers are unique to each sampling event and grid/area and include the Site, grid identifier, and date of the sampling event. (Note that when a CVS sample represented a 1-acre area (i.e., group of four ¼-acre grids), for simplicity the sample identifier included only 1 of the 4 grid identifiers.) CVS results that indicate an RG failure are distinguished by a boxed or bolded entry. Italicized entries indicate that the CVS result was less than the method detection limit, in which case the presented value is the method detection limit. The TEQ entry is shaded for grids that failed to achieve the dioxin/furan RG. Shaded columns indicate that an excavation event occurred between two CVS sampling events; the excavation depth is indicated in the column heading.

Each dioxin/furan measurement is expressed as a calculated 2,3,7,8 TCDD TEQ in a row near the bottom of the table. Calculation of the 2,3,7,8 TCDD TEQ is detailed in Section 2.1. The data presented in the October 7, 2011 database and summarized in Appendix A is of acceptable quality and decisively usable, and it establishes that all dioxin/furan sampling areas have attained the dioxin/furan RG.

Section 5. Conclusion

The 2002 Krejci Site Record of Decision (ROD) requires, among other things, that all debris and soils containing unacceptable levels of contaminants will be excavated and disposed off-site at appropriately licensed or permitted facilities. Site remediation goals (RGs) for each of thirty nine (39) identified Site contaminants were established in the ROD and incorporated into the 2002 Consent Decree with Ford Motor Company (Ford). The Consent Decree established that post-excavation soil sampling and testing would be performed to verify that remaining soils achieve the Site RGs. To that end, a rigorous soil sampling, testing, and cleanup verification program was designed on behalf of Ford, approved by NPS, and documented in the Cleanup Verification Sampling (CVS) Plan (including the Quality Assurance Project Plan (QAPP)), Appendix C of the Remedial Design (RD) Report (2005).

Excavation of contaminated Site material pursuant to the ROD and Consent Decree began in October, 2005 and continued in phases through August, 2011. The CVS program was implemented by EQIS, on behalf of Ford, during and following the period of Site excavation. EQIS validated the CVS data prior to entering it into the CVS database, and then divided the data into three (3) data sets (on a chronological basis) to perform its data quality evaluation. EQIS documented its data evaluation in three (3) Data Quality Evaluation (DQE) reports, each of which pertains to a different data set. The DQE reports concluded that the data in the CVS database is acceptable and usable to determine achievement of Site RGs. The DQE reports also concluded that all Site RGs have been achieved.

NPS commissioned data usability reports (DURs) to correspond with the DQEs to enable NPS's independent evaluation of EQIS's conclusion that the CVS data is usable and sufficient to establish that

all Site RGs have been achieved for all contaminants as required in the Consent Decree. This DUR 3 is the third and last of such reports. These DURs document a thorough and comprehensive review and evaluation of all CVS data, Site records, compliance with QAPP data quality objectives (DQOs), implementation of the CVS analytical program, audit reports, procedures set forth in the QAPP, and laboratory quality controls. The three DURs, taken together, support the following conclusions:

- The Krejci Site cleanup verification soil sampling and testing program was implemented according to the agreed procedures and resulted in quality data that may be used to determine whether the Site RGs have been achieved.
- Data results of the Krejci Site cleanup verification soil sampling and testing program establish that all Site remediation goals have been achieved for all Site contaminants in all grids throughout the Site as required in the Consent Decree.

Section 6 Related References

June 2000, Final Remedial Investigation Report, Krejci Dump Site, Cuyahoga Valley National Recreation Area, prepared for the National Park Service by the Bureau of Reclamation, Lakewood CO.

April 2002, Partial Consent Decree, United States v. Chrysler Corp., et al., Civil Action No. 5:97 CV00894 (N.D. Ohio).

June 2005, Final (100%) Remedial Design Report, Krejci Dump Site, Cuyahoga Valley National Park, Summit County, Ohio, prepared for EQIS, Ypsilanti, MI, by Conestoga-Rovers & Associates, Waterloo, Ontario.

September 2005, Remedial Action Work Plan, Krejci Dump Site, Cuyahoga Valley National Park, Summit County, Ohio, prepared for EQIS, Ypsilanti, MI, by Conestoga-Rovers & Associates, Waterloo, Ontario.

July 2009, Amendments to the Krejci Site Remedial Action Documents (effective as of June 8, 2009).

May 2012, Data Quality Evaluation 1, Cleanup Verification Sampling Results For August 2008 to September 2009, prepared for EQIS, Ypsilanti, MI, by ReSolution Partners, LLC, Madison, Wisconsin.

May 2012, Data Quality Evaluation 2, Cleanup Verification Sampling Results For October 2009 to December 2010, prepared for EQIS, Ypsilanti, MI, by ReSolution Partners, LLC, Madison, Wisconsin.

May 2012, Data Quality Evaluation 3, Cleanup Verification Sampling Results For January 2011 to December 2011, prepared for EQIS, Ypsilanti, MI, by ReSolution Partners, LLC, Madison, Wisconsin.

May 2012, Data Usability Report 1, Cleanup Verification Sampling Data, Krejci Dump Site, prepared for the National Park Service, by MCG Geotechnical Engineering Inc., Morrison, Colorado.

May 2012, Data Usability Report 2, Cleanup Verification Sampling Data, Krejci Dump Site, prepared for the National Park Service, by MCG Geotechnical Engineering Inc., Morrison, Colorado.

Appendix A

CVS and Comparisons to Remediation Goals