

**REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
FOR
MANY DIVERSIFIED INTERESTS SUPERFUND SITE
HOUSTON, TEXAS**

**DATA EVALUATION SUMMARY REPORT
ON-SITE REMEDIAL INVESTIGATION**

Prepared for

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Work Assignment No.	:	107-RICO-06CN
EPA Region	:	6
Date Prepared	:	October 3, 2003
Revision No.	:	00
Contract No.	:	68-W6-0037
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ACRONYMS AND ABBREVIATIONS

µg/L	Microgram per liter
Accutest	Accutest Laboratories, Inc.
CLP	Contract Laboratory Program
COPC	Contaminant of potential concern
CRDL	Contact-required detection limit
CRQL	Contract-required quantitation limit
DESR	Data evaluation summary report
DQO	Data quality objective
DVR	Data validation report
e-Lab	e-Lab, Inc.
EPA	U.S. Environmental Protection Agency
FAA	Furnace atomic absorption
FSP	Field sampling plan
GC	Gas chromatography
GC/MS	Gas chromatography/mass spectrometer
HHRA	Human health risk assessment
ICP	Inductively-coupled plasma
IDL	Instrument detection limit
IDW	Investigation-derived waste
LCS	Laboratory control sample
MD	Matrix duplicate
MDI	Many Diversified Interests
MDL	Method detection limit
MS	Matrix spike
MSD	Matrix spike duplicate
PARCCS	Precision, accuracy, representativeness, completeness, comparability, and sensitivity
PCB	Polychlorinated biphenyl
PE	Performance evaluation
Pest	Pesticide
QA	Quality assurance
QAPP	Quality assurance project plan
QC	Quality control
RAC	Response Action Contract
RCI	Reactivity, corrosivity, and ignitability
RI	Remedial investigation
RPD	Relative percent difference
RRF	Relative response factor
RSD	Relative standard deviation
SDG	Sample delivery group
SLERA	Screening level ecological risk assessment
SQL	Sample quantitation limit
SVOC	Semivolatile organic compound
TAL	Target Analyte List
TCEQ	Texas Commission on Environmental Quality
TCL	Target Compound List
TCLP	Toxicity characteristic leaching procedure
Tetra Tech	Tetra Tech EM Inc.
VOC	Volatile organic compound
WRS	Wilcoxon rank sum
XRF	X-ray fluorescence

1.0 INTRODUCTION

This data evaluation summary report (DESR), prepared by Tetra Tech EM Inc. (Tetra Tech), documents and summarizes analytical data collected during the on-site remedial investigation (RI) at the Many Diversified Interests (MDI) Superfund Site located in Houston, Harris County, Texas conducted under the U.S. Environmental Protection Agency (EPA) Region 6 Response Action Contract (RAC). The purpose of this document is to summarize analytical data quality and usability as related to the project-specific data quality objectives (DQO) stated in the quality assurance project plan (QAPP) prepared for the MDI Site (Tetra Tech 2003b). The DQO process is a series of planning steps designed to ensure that the type, quantity, and quality of environmental data used in decision-making are appropriate for the intended application. Overall DQOs for the remedial investigation at MDI were developed and presented in the QAPP (Tetra Tech 2003b). The principal study question for the MDI site derived from the DQO process is as follows:

Where do levels of preliminary contaminants of potential concern (COPC) exist either on or off site at concentrations above risk-based screening levels and/or background mean concentrations along complete exposure pathways for relevant exposure scenarios?

Secondary study questions are as follows:

1. Where do preliminary COPC concentrations in Phase I media exceed human health risk-based screening levels?
2. Where do preliminary COPC concentrations in Phase I media exceed ecological risk-based screening levels?
3. What are the potential migration and exposure pathways for these contaminants, and is there a site-related gradient moving away from the MDI site that links preliminary COPCs to a site release?

In order to address these study questions, a sampling and analysis plan was designed to support all data requirements. Other objectives of this investigation were to (1) determine a suitable remedial alternative for cleanup if COPCs were detected at concentrations requiring cleanup and (2) characterize waste materials at the site for proper disposal selection.

For the on-site investigation (discussed in this DESR), a total of 1,005 soil samples were analyzed using field-based x-ray fluorescence (XRF), while 161 soil samples, 35 temporary well ground water samples, 24 permanent monitoring well ground water samples, 1 surface water sample, and 8 sediment samples

were submitted for off-site laboratory analysis. In addition to the investigative samples listed above, 18 remedy samples and 25 samples of various waste materials were collected and submitted for analysis. All samples were analyzed by numerous Contract Laboratory Program (CLP) laboratories, the EPA Region 6 laboratory, and two Tetra Tech contractor laboratories (e-Lab, Inc. [e-Lab] and Accutest Laboratories, Inc. [Accutest] of Houston, Texas).

Based on the historical operations and waste management practices at the former metal casting foundry, investigative multimedia samples collected during the on-site RI were analyzed for metals (including molybdenum), volatile organic compounds (VOC), semivolatile organic compounds (SVOC), and pesticides/polychlorinated biphenyls (PCB).

Soil, temporary well ground water, permanent monitoring well ground water, sediment, surface water, and waste samples were collected between February 2003 and May 2003. On-site samples were collected from within the site boundaries at the MDI site located at 3617 Baer Street in Houston, Texas. An additional off-site residential soil sampling investigation was undertaken as part of this same work assignment; however, the data summary will be presented in a separate document. This DESR provides a general overview of analytical data quality for the on-site sampling event, including the following discussions:

- Data validation (guidelines, responsibilities, and analytical findings)
- Evaluation of laboratory and field-based data
- Data evaluation parameters
- DQO evaluation
- Background data (statistics and results)
- Conclusions

Data were validated and evaluated based on EPA guidance documents and against project-specific DQOs. The data validation guidelines, responsibilities, and results are presented in Section 2.0 of this DESR. Section 3.0 provides a summary of the statistical analysis of analytical data as it relates to the comparison of fixed-laboratory inductively coupled plasma (ICP) metals results to field-based XRF metals results. Acceptability of data, evaluated by the parameters of precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS), is determined through the process of data validation. Specific PARCC parameters are discussed in Section 4.0 of this DESR. Data usability with

regard to the DQOs is presented in Section 5.0, which includes a discussion of general quality control (QC) issues. The project team developed DQOs that are discussed in Section 5.0 of this DESR and described in the QAPP for the MDI Site (Tetra Tech 2003b). Methods and techniques required to yield analytical data of acceptable quality and quantity to support DQOs are also outlined in the QAPP. Section 5.0 also discusses data usability with regard to risk assessment, and presents the comparison of expected reporting limits to actual reporting limits and their impact on risk assessments. Section 6.0 discusses background metals data obtained off site for comparison to on-site data. The conclusions regarding the data evaluation are presented in Section 7.0.

2.0 DATA VALIDATION

This section describes the data validation guidelines, responsibilities, and QC findings for each type of analytical data provided by supporting laboratories. Field samples were collected and sent to three types of laboratory facilities: (1) EPA CLP laboratories, (2) EPA Region 6 Laboratory, and (3) Tetra Tech subcontractor laboratories. Data were submitted to Tetra Tech in sample delivery groups (SDG) from each of the laboratories. According to the requirements of the QAPP, data from EPA CLP laboratories and the Region 6 laboratory were submitted to Tetra Tech post validation, and required no further validation by Tetra Tech. Electronic deliverables submitted to Tetra Tech from EPA CLP laboratories and the Region 6 laboratory contained suitable data validation qualifiers and accompanying data validation summaries. Data validation qualifiers and definitions used for CLP data are presented in Table 1. The only laboratory qualifier used by the EPA Region 6 laboratory for data review was a “U.” The “U” qualifier means that an analysis was conducted on the analyte, but the analyte was undetected by the instrument; the reported numerical value is the quantitation limit for the sample. Attachment A of this DESR contains the data validation reports (DVR) for each of the 71 SDGs submitted by EPA CLP laboratories, and Attachment B of this DESR contains the data report narratives accompanying each of the six SDGs from EPA Region 6 laboratory.

Tetra Tech also subcontracted two non-CLP laboratories (Accutest of Houston, Texas, and e-Lab of Houston, Texas) to provide analytical support for the analysis of molybdenum in a small portion of off-site samples (not included in this DESR), waste characterization samples (from on-site investigation), and remedy suite analyses for on-site samples. A complete listing of analyses is presented in the project-specific QAPP (Tetra Tech 2003b). Data for molybdenum analysis, as well as waste characterization and remedy suites provided by non-CLP subcontractor laboratory were validated by Tetra Tech chemists

TABLE 1
CLP DATA VALIDATION QUALIFIERS

Qualifier	Definition
Organic Analysis Data Validation Qualifiers	
U	Not detected at reported quantitation limit
N	Identification is tentative
J	Estimated value
L	Reported concentration is below the CRQL
M	Reported concentration should be used as a raised quantitation limit because of interferences and/or laboratory contamination
R	Unusable
^	High biased; actual concentration may be lower than the concentration reported
v	Low biased; actual concentration may be higher than the concentration reported
F+	A false positive
F-	A false negative
B	This result may be high biased because of laboratory/field contamination. The reported concentration is above 5 or 10 times the concentration reported in the method/field blank.
UJ	Estimated quantitation limit
T	Identification is questionable because of absence of other commonly coexisting pesticides
*	Results not recommended for use because of associated QA/QC performance inferior to that from other analysis
W	The result should be used with caution. The result was reported on a dry weight basis although the sample did not conform to the EPA Office of Water definition of a soil sample because of its high water content (greater than 70 percent moisture).
Inorganic Analysis Data Validation Qualifiers	
U	Undetected at the laboratory reported detection limit (IDL)
L	Reported concentration is between the IDL and the CRDL
J	Result is estimated because of outlying quality control parameters such as matrix spike, serial dilution, FAA spike recovery, etc.
R	Result is unusable
F	A possibility of a false negative exists
UC	Reported concentration should be used as a raised detection limit because of apparent blank contamination
^	High bias; actual concentration may be lower than the concentration reported
v	Low bias; actual concentration may be higher than the concentration reported
W	The result should be used with caution. The result was reported on a dry weight basis although the sample did not conform to the EPA Office of Water definition of a soil sample because of its high water content (greater than 70 percent moisture).

Notes:

CLP	Contract Laboratory Program	FAA	Furnace atomic absorption
CRDL	Contract-required detection limit	IDL	Instrument detection limit
CRQL	Contract-required quantitation limit	QA/QC	Quality assurance/quality control
EPA	U.S. Environmental Protection Agency		

in accordance with the following EPA guidance documents, “USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review” (EPA 2002a) and “USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review” (EPA 1999b). In addition to EPA validation guidance documents, Tetra Tech used acceptance criteria presented in the QAPP and analytical methods for validation of non-CLP results. Data validation qualifiers and definitions used for non-CLP data are based on EPA National Functional Guidelines documents (EPA 1999b and 2002a) and are presented in Table 2. Appendix A of this DESR contains the DVRs for each of the six SDGs from Tetra Tech’s non-CLP laboratories. In preparing this DESR, Tetra Tech reviewed all DVRs and cover letters (from EPA, as well as Tetra Tech reviewers) and summarized the findings in the following sections. The QC findings by analytical method are presented in the subsections. The findings discussed in the following sections address only those issues that resulted in qualification of data. Other minor findings that were deemed insignificant to data quality are discussed in individual DVRs included in the appendices. A complete listing of all analytical methods is presented in the QAPP (Tetra Tech 2003b).

2.1 CLP VOLATILE ORGANIC COMPOUNDS

CLP Target Compound List (TCL) VOC analyses were performed according to the CLP analytical protocols “EPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration (OLM04.2)” (EPA 1999a) and “EPA Contract Laboratory Program Statement of Work for Low Concentration Organic Analysis, Multi-Media, Multi-Concentration (OLC03.2)” (EPA 2000b), by EPA CLP laboratories. TCL VOC analyses were conducted on the following quantities and types of field and QC samples:

- 35 temporary well ground water samples (plus 4 sets of field duplicates and 3 sets of matrix spikes [MS] and matrix spike duplicates [MSD])
- 24 monitoring well ground water samples (plus 3 sets of field duplicates and 2 sets of MS/MSDs)
- 109 soil samples (plus 13 sets of field duplicates and 9 sets of MS/MSDs)
- 1 surface water sample (plus 1 set of field duplicates and 1 set of MS/MSDs)
- 21 trip blanks
- 3 equipment rinsates

TABLE 2

EPA FUNCTIONAL GUIDELINES DATA VALIDATION QUALIFIERS

Qualifier	Definition
Organic Analysis Data Validation Qualifiers	
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a “tentative identification.”
NJ	The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
Inorganic Analysis Data Validation Qualifiers	
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimate quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

Note:

EPA U.S. Environmental Protection Agency

Data validation findings are summarized in the following paragraphs.

Holding Times and Preservation – The 14-day analytical holding time requirement for ground water, soil, and surface water was met for all samples. Results for aromatic compounds in ground water samples and trip blanks were qualified as estimated and possibly biased low (UJv), because the pH of the samples was greater than 2 units upon receipt at the laboratory. The reviewer indicated that reported results for aromatic compounds in these samples might be lower than actual concentrations in the matrix. It was noted by the field sampling teams that several of the water samples effervesced slightly as the bottles were being filled.

Instrument Performance – Gas chromatograph/mass spectrometer (GC/MS) instruments met the performance criteria for the tuning check compound. No data were qualified on the basis of instrument performance.

Instrument Calibration and Calibration Checks – Initial and continuing calibrations were performed at the proper concentrations and frequencies. The quantitation limits for 1,2-dibromo-3-chloropropane in five soil samples (JS14-0.5-1.5, JS17-0.5-1.5, JS17-16.0-18.0, JS18-0.5-1.5, and JS18-12.5-14.5) were qualified as unusable (R), because the technical minimum relative response factor (RRF) criterion was not met for calibrations associated with these samples. Results for methyl acetate in two surface water samples (SW-02 and SW-02D), four monitoring well ground water samples (MW-03-01, MW-08-01, MW17-01, and MW18-01), and two trip blanks (TB-17 and TB-18) were also qualified as unusable (R), because the RRF exceeded the technical criterion in the initial calibration associated with these samples. Rejected results are not usable to meet DQOs because calibration responses were too low for accurately quantifying sample concentrations.

Results for bromochloromethane in ground water samples were qualified as estimated and biased low (Jv) because the RRF exceeded the technical criterion in the initial calibration associated with these samples. The quantitation limits for methyl acetate in a trip blank and equipment rinsate blank were qualified as estimated and biased low (UJv) because the RRF exceeded the technical criterion in the initial calibration associated with these samples.

Results for acetone in ground water samples and a trip blank were qualified as estimated (J) because the percent relative standard deviation (RSD) exceeded the technical criterion for the initial calibration associated with these samples. Acetone and bromoform results in soil samples, ground water samples,

and trip blanks were qualified as estimated (J) because the percent difference exceeded the technical criterion for the continuing calibration associated with these samples.

Results for 4-methyl-2-pentanone in soil samples were qualified as estimated (J) because the 2-hexanone chromatographic peak was misidentified as 4-methyl-2-pentanone in the calibrations associated with these samples.

Method Blanks – Acetone, bromoform, bromomethane, 2-butanone, carbon disulfide, chloromethane, 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, dichlorodifluoromethane, styrene, cis-1,3-dichloropropene, trans-1,3-dichloropropene, ethylbenzene, isopropylbenzene, methylene chloride, 4-methyl-2-pentanone, tetrachloroethene, toluene, 1,2,3-trichlorobenzene, 1,2,4-trichlorobenzene, trichloroethene, and xylenes were detected in laboratory method blanks. As such, many sample results were qualified because of this contamination. Results for the compounds listed above were qualified as undetected (U) in associated samples that had concentrations less than the CRQL. The results for associated samples were qualified as undetected with an elevated quantitation limit (UM), when the concentration was above the sample quantitation limit (SQL). Other results in associated samples were qualified as estimated (J or B) when concentrations were greater than the action levels for blank qualification (5 or 10 times the blank concentration), because the results may be biased high due to laboratory contamination. If the contaminant compounds were not detected in associated samples, then no qualification was applied. Common laboratory contaminants are compounds that are frequently used in the laboratory for purposes other than calibration standards (usually as solvents) and include methylene chloride, acetone, 2-butanone, and cyclohexane. Since the list of contaminants found in method blanks for this project is quite extensive, the source for these contaminants was discussed in the DVRs. The data reviewer indicated that the presence of compounds in the laboratory storage blank might indicate that cross-contamination of these compounds may have occurred during sample storage. Three other possible causes may account for the excessive method blank contamination: (1) contamination of laboratory dilution/purge water, (2) internal carryover of contaminants between standards and samples in the instrument, or (3) external carryover of contaminants from the laboratory work areas to the instrument. The source of the contamination was not determined.

Trip Blanks – A total of 21 trip blanks were prepared, shipped, and analyzed with field samples. VOCs were detected in trip blanks that accompanied field samples to the laboratories. VOCs were detected in one or more trip blanks at the maximum concentration listed in Table 3.

TABLE 3
TRIP BLANK CONTAMINANT CONCENTRATIONS

Contaminant	Concentration ($\mu\text{g/L}$)
Benzene	0.035
Carbon disulfide	0.051
Chloroform	0.8
Chloromethane	0.16
Cyclohexane	0.10
1,4-Dichlorobenzene	0.057
trans-1,2-Dichloroethene	0.057
cis-1,3-Dichloropropene	0.11
Ethylbenzene	0.14
4-Methyl-2-pentanone	2.0
Methylene chloride	2.0
Methyl-tert-butyl ether	0.16
Styrene	0.1
Tetrachloroethene	0.39
Trichloroethene	0.8
Trichlorofluoromethane	0.057
Toluene	6.0

Note:

$\mu\text{g/L}$ Microgram per liter

Based on the concentrations of contaminants detected in trip blanks, data were qualified as undetected (U) in associated samples that had concentrations less than the CRQL. Results were qualified as undetected with an elevated quantitation limit (UM) when the concentration was above the SQL. Other results were qualified as estimated (J or B) when concentrations were greater than the action levels for blank qualification, because the results may be biased high due to blank contamination. If the contaminant compounds were not detected in associated samples, then no qualification was applied.

The amount of contaminants detected in trip blanks might indicate that cross-contamination occurred during shipment of samples from the field to the laboratory. However, when compared with the laboratory method blank contaminants discussed in the preceding section, trip blank contamination might have occurred in the laboratory (as is suspected with the method blanks).

Equipment Rinsate Blanks – Three equipment rinsate blanks were collected and analyzed for VOCs to measure the effectiveness of sampling equipment decontamination procedures. Six VOCs were detected in the equipment rinsate blanks at the maximum concentrations indicated: benzene at 0.038 micrograms per liter ($\mu\text{g/L}$), bromoform at 0.088 $\mu\text{g/L}$, chloroform at 0.7 $\mu\text{g/L}$, chloromethane at 0.068 $\mu\text{g/L}$, cis-1,3-dichloropropene at 0.091 $\mu\text{g/L}$, and toluene at 5.0 $\mu\text{g/L}$. Since all of these compounds are also detected in method blanks and/or trip blanks, no real conclusion about sampling equipment decontamination can be drawn. The source of equipment rinsate contamination cannot be determined.

System Monitoring Compounds/Surrogates – Appropriate surrogate compounds were spiked into samples, and percent recoveries were evaluated. Sample results for 4-methyl-2-pentanone and 2-hexanone in sample TB-17 were qualified as rejected (UR or R) because of very low surrogate recoveries. Results for 1,1,2,2-tetrachloroethane, 1,1-dichloroethane, 1,2-dibromo-3-chloropropane, 1,2-dibromoethane, bromochloromethane, bromoform, and dibromochloromethane in sample TWH6-01 were rejected (UR) due to very low surrogate recoveries. Very low surrogate recoveries indicate that the efficiency of purging VOCs from the samples to the instrument was not adequate or that matrix interference prohibited the accurate quantitation of surrogate compounds. In either case, the potential for false negatives exists, and data are unusable.

Due to low surrogate recoveries, sample results for 2-hexanone, 4-methyl-2-pentanone, benzene, 1,1-dichloroethane, cis-1,2-dichloroethene, trans-1,2-dichloroethene, cis-1,3-dichloropropene, trans-1,3-dichloropropene, bromochloromethane, chloroform, and 1,1,1-trichloroethene were qualified as estimated and biased low (UJv or Jv). Low surrogate recoveries might indicate that the efficiency of purging VOCs from the samples to the instrument was not adequate or that matrix interference prohibited

the accurate quantitation of surrogate compounds. In either case, the potential for biased low results exists, and data are considered estimated.

Matrix Spike and Matrix Spike Duplicate – MSs and MSDs were analyzed with field samples according to the CLP protocol. Percent recoveries and relative percent differences (RPD) were within QC limits and required no qualification of sample results.

Laboratory Control Sample – Laboratory control samples (LCS) were required only for analysis of ground water and surface water samples conducted under the low concentration CLP protocol (EPA 2000). LCSs were prepared and analyzed at the required frequencies. Percent recoveries were within QC limits and required no qualification of sample results.

Internal Standards – Internal standard area counts and retention times were within QC limits. No qualification of sample results was required on this basis.

Compound Identity and Quantitation – No compound identification or quantitation issues were identified for VOC analysis. However, compound results were frequently reported below CRQLs. Detected results reported below the CRQL were qualified as estimated (LJ). Quantitation limits may be greater than project-required quantitation limits due to dilutions required when compound responses exceed calibration ranges and when moisture content is high.

Field Duplicates – Thirteen pairs of field duplicates were analyzed for soil samples, four pairs for temporary well ground water samples, three pairs for monitoring well ground water samples, and one pair for surface water samples. The precision of detected results was calculated and evaluated against the QC criterion. Inconsistent field duplicate results, (primarily for soil samples) were identified and are indicative of the heterogeneity of the soil matrix and uneven distribution of contaminants in the medium. Results for acetone and bromoform were qualified as estimated (J) due to inconsistencies in field duplicate results.

2.2 CLP SEMIVOLATILE ORGANIC COMPOUNDS

CLP TCL SVOC analyses were performed according to the CLP analytical protocol “EPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration (OLM04.2)” (EPA 1999a) and “EPA Contract Laboratory Program Statement of Work for Low Concentration Organic Analysis, Multi-Media, Multi-Concentration (OLC03.2)” (EPA 2000b), by EPA

CLP laboratories. TCL SVOC analyses were conducted on the following quantities and types of field and QC samples:

- 24 monitoring well ground water samples (plus 3 sets of field duplicates and 2 sets of MS/MSDs)
- 193 soil samples (plus 22 sets of field duplicates and 12 sets of MS/MSDs)
- 1 surface water sample (plus 1 set of field duplicates and 1 set of MS/MSDs)
- 8 sediment samples (plus 1 set of field duplicates and 1 set of MS/MSDs)
- 3 equipment rinsates

Data validation findings are summarized in the following paragraphs.

Holding Times and Preservation – The 14-day extraction and 40-day analytical holding time requirements for soil and sediment samples were met. The 7-day extraction and 40-day analytical holding time requirements for ground water and surface water samples were met.

Instrument Performance – GC/MS instruments met the performance criteria of the tuning check compound. No data were qualified on the basis of instrument performance.

Instrument Calibration and Calibration Checks – Initial and continuing calibrations were performed at the proper concentrations and frequencies. Initial calibration criteria for RSDs and RRFs were generally met. However, quantitation limits for 2,4-dinitrophenol in 16 soil samples were qualified as unusable (R), because the technical minimum RRF criterion was not met for calibrations associated with these samples. These 16 soil samples are: JS45-0.5-1.5, JS45-19.5-20.2, JS46-0.0-0.5, JS46-0.5-1.5, JS46-11.0-12.0, JS49-0.0-0.5, JS49-0.5-1.5, JS49-14.0-16.0, JS47-0.0-0.5, JS47-0.5-1.5, JS47-15.0-16.0, JS48-0.0-0.5, JS48-15.0-16.0, JS54-0.0-0.5, JS54-0.5-1.5, and JS54-14.0-15.0. Rejected results are not usable to meet DQOs, because calibration responses were too low to accurately quantify sample concentrations.

The fluorene result in one soil sample was qualified as estimated (J) because the percent RSD criteria was not met in the associated initial calibration. The di-n-octylphthalate result in one soil sample was qualified as estimated (J) because the percent difference exceeded the technical criterion for the continuing calibration associated with this sample.

Method Blanks – Acetophenone, benzaldehyde, di-n-butylphthalate, bis(2-ethylhexyl)phthalate, phenol, and benzo(a)pyrene were detected in laboratory method blanks. Phthalates are typically considered common laboratory contaminants. As such, associated sample results were qualified because of this contamination. Results for the compounds listed above were qualified as undetected (U) in associated samples that had concentrations less than the CRQL. Results were qualified as undetected with an elevated quantitation limit (UM) when the concentration was above the SQL. Other results in associated samples were qualified as estimated (J or B) when concentrations were greater than the action levels for blank qualification, because the results may be biased high due to laboratory contamination. If the contaminant compounds were not detected in associated samples, then no qualification was applied.

Equipment Rinsate Blanks – Three equipment rinsate blanks were collected and analyzed for SVOCs to measure the effectiveness of sampling equipment decontamination procedures. Naphthalene was the only SVOC detected in an equipment rinsate blanks at the maximum concentration of 0.3 µg/L. Naphthalene was not detected in laboratory method blanks, so its source is probably field-related activities. The concentration of naphthalene in the equipment rinsate blanks is at trace level, and poses little if any effect on sample results or overall data quality. As such, no data were qualified.

System Monitoring Compounds/Surrogates – Appropriate surrogate compounds were spiked into samples, and percent recoveries were evaluated. Quantitation limits for benzo(b)fluoranthene, benzo(a)pyrene, and dibenzo(a,h)anthracene were qualified as rejected (UR) due to extremely low surrogate recoveries (less than 10 percent recovery) in sample MW-13-01. Quantitation limits for the following compounds in the samples listed were also qualified as rejected (UR) due to extremely low surrogate recoveries (less than 10 percent recovery).

- 3,3'-Dichlorobenzidine, 4-chloroaniline, and hexachlorocyclopentadiene in samples MW-05-01 and MW15-01
- 4-Chloroaniline, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, dibenzo(a,h)anthracene, hexachlorocyclopentadiene, and indeno(1,2,3-cd)pyrene in sample MW-13-01

Very low surrogate recoveries indicate that the efficiency of extracting SVOCs from the sample matrix to the solvent was not adequate or that matrix interference prohibited the accurate quantitation of surrogate compounds. In either case, the potential for false negatives exists, and data are unusable.

Due to low surrogate recoveries, results for benzo(a)anthracene, benzo(b)fluoranthene, benzo(a)pyrene, and dibenzo(a,h)anthracene were qualified as estimated and biased low (Jv) in affected samples. In addition, quantitation limits for 4,6-dinitro-2-methylphenol, 2-methylphenol, 4-methylphenol,

2,4-dimethylphenol, hexachlorobenzene, atrazine, phenanthrene, anthracene, fluoranthene, pyrene, benzo(a)anthracene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, chrysene, indeno(1,2,3-cd)pyrene, dibenzo(a,h)anthracene, benzo(g,h,i)perylene, caprolactam, 1,1'-biphenyl, dimethylphthalate, diethylphthalate, di-n-butylphthalate, butylbenzylphthalate, di-n-octylphthalate, and bis(2-ethylhexyl)phthalate in soil samples were qualified as estimated and biased low (UJv) because of low surrogate recoveries in the affected samples.

Matrix Spike and Matrix Spike Duplicate – MSs and MSDs were analyzed with field samples according to the CLP protocol. Percent recoveries and RPDs were generally within QC limits and required no qualification of sample results. However, the quantitation limit for 4-nitrophenol in sample SD-01 was qualified as rejected (UR) because of zero percent recoveries in the MS and MSD analyses. When a spiking compound is not recovered in both the MS and MSD, it usually indicates that matrix interference exists that masks the ability to quantify the spiking compound. If such a condition exists, then the accurate quantification of a target compound is equally difficult. False negatives are possible; therefore, the data are unusable.

Laboratory Control Sample – LCSs were required only for analysis of ground water and surface water samples conducted under the low concentration CLP protocol (EPA 2000b). LCSs were prepared and analyzed at the required frequencies. Percent recoveries were within QC limits and required no qualification of sample results.

Internal Standards – Internal standard retention times were within QC limits; however, samples with matrix interferences did not meet response area count criteria. Samples that initially did not meet QC criteria were diluted in an attempt to reduce the matrix effect on the chromatogram. However, when subsequent dilution analyses also failed QC criteria, positive results for SVOC compounds (including bis[2-ethylhexyl]phthalate, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, phenanthrene, anthracene, fluoranthene, pyrene, carbazole, indeno[1,2,3-cd]pyrene, dibenzo[a,h]anthracene, chrysene, and benzo[a]anthracene) were qualified as estimated and biased low (Jv). Quantitation limits for atrazine, 4,6-dinitro-2-methylphenol, 4-bromophenyl phenylether, N-nitrosodiphenylamine, hexachlorobenzene, pentachlorophenol, phenanthrene, anthracene, carbazole, di-n-butylphthalate, fluoranthene, pyrene, butylbenzylphthalate, 3,3'-dichlorobenzidine, benzo(a)anthracene, bis(2-ethylhexyl)phthalate, di-n-octylphthalate, benzo(b)fluoranthene, benzo(k)fluoranthene, indeno(1,2,3-cd)pyrene, dibenzo(a,h)anthracene, and benzo(g,h,i)perylene were qualified as estimated and biased low (UJv) because internal standard responses were low and compounds were not detected in affected samples.

Compound Identity and Quantitation – No compound identification or quantitation issues were identified for SVOC analysis, except that benzo(b)fluoranthene and benzo(k)fluoranthene could not be chromatographically resolved by the GC/MS in some samples. In these cases, sample results for both isomers were qualified as estimated and biased high (J[^]) because the value reported is possibly the sum of both isomers. Dilutions analyzed as a result of matrix interference elevated the quantitation limit by the dilution factor.

Compound results were frequently reported below CRQLs. Detected results reported below the CRQL were qualified as estimated (LJ). Quantitation limits may be greater than project-required quantitation limits due to dilutions required when compound responses exceed calibration ranges and when moisture content is high.

For sediment samples (SD-02 and SD-01), SVOC compounds were qualified with a cautionary flag (W) to indicate that the method of analysis (soil method) might be ineffective for analyzing samples with moisture contents in excess of 70 percent. While the detected compounds are confirmed with mass spectral data, the values are estimates since the analytical method is not optimal for the matrix.

Field Duplicates – Twenty-two pairs of field duplicates were analyzed for soil samples, three pairs for monitoring well ground water samples, one pair for surface water samples, and one pair for sediment samples. The precision of detected results was calculated and evaluated against the QC criterion. Inconsistent field duplicate results, (primarily for soil samples) were identified and are indicative of the heterogeneity of the soil matrix and uneven distribution of contaminants in the medium. As such, results for bis(2-ethylhexyl)phthalate, benzo(g,h,i)perylene, cabazole, phenanthrene, anthracene, pyrene, chrysene, benzo(a)anthracene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenzo(a,h)anthracene, fluoranthene, and benzo(b)fluoranthene were qualified as estimated (J) in field duplicates because of inconsistent results between duplicate pairs.

2.3 CLP PESTICIDES AND POLYCHLORINATED BIPHENYLS

CLP TCL pesticide and PCB analyses were performed according to the CLP analytical protocol “EPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration (OLM04.2)” (EPA 1999a), by EPA CLP laboratories. TCL pesticide/PCB analyses were conducted on the following quantities and types of field and QC samples:

- 124 soil samples (plus 14 sets of field duplicates and 12 sets of matrix spikes)

- 8 sediment samples (plus 1 set of field duplicates and 1 set of matrix spikes)
- 2 equipment rinsates

Data validation findings are summarized in the following paragraphs.

Holding Times and Preservation – The 14-day extraction and 40-day analytical holding time requirements for soil and sediment samples were met.

Instrument Performance – Gas chromatograph (GC) instruments met performance criteria. No data were qualified on the basis of instrument performance.

Instrument Calibration and Calibration Checks – Initial and continuing calibrations were performed on the gas chromatograph (GC) at the proper concentrations and frequencies. All technical criteria were met, and no data were qualified based on calibration criteria.

Method Blanks – Method and instrument blanks met technical criteria; no target analytes were detected in associated blanks.

Equipment Rinsate Blanks – Two equipment rinsate blanks were collected and analyzed for pesticides/PCBs to measure the effectiveness of sampling equipment decontamination procedures. No target analytes were detected in equipment rinsate blanks.

System Monitoring Compounds/Surrogates – Appropriate surrogate compounds were spiked into samples, and percent recoveries were evaluated. Positive results for Aroclor 1254, Aroclor 1260, and endrin ketone were qualified as estimated and biased high (J[^]) in samples due to high recoveries of surrogate compounds.

Matrix Spike and Matrix Spike Duplicate – MSs and MSDs were analyzed with field samples according to the CLP protocol. Percent recoveries and RPDs were generally within QC limits and required no qualification of sample results. However, quantitation limits for heptachlor, γ -BHC, endrin, and 4,4'-DDT were qualified as estimated and biased low (UJv) due to low MS and MSD recoveries in one soil sample.

Laboratory Control Sample – LCSs are not required for the pesticide/PCB protocol.

Internal Standards – Internal standards are not applicable to pesticide/PCB analyses by GC.

Compound Identity and Quantitation – Results for numerous detected pesticides and Aroclors were qualified as estimated (J) because the percent difference between values reported from each of the two GC columns varied by more than 25 percent. Results for endosulfan I, γ -chlordane, and Aroclor 1260 were qualified as tentatively identified (N) because of suspected interference from the presence of Aroclor 1260 in the soil samples. Identification of endosulfan I, β -BHC, γ -chlordane, 4,4'-DDD, 4,4'-DDE, and endrin ketone in several soil samples were questionably qualified (T) because of the absence of commonly co-existing pesticides (endosulfan II, other BHCs, other chlordanes, DDT products, and other endrins). Results for 4,4'-DDE were qualified as undetected (U) in samples after the review indicated that the initially detected result was actually a component peak from Aroclor 1254. Quantitation limits for dieldrin, endosulfan sulfate, 4,4'-DDT, endrin ketone, endrin aldehyde, and γ -chlordane in soil samples were qualified as having elevated quantitation limits (UM) because aroclor peaks created an interference with these compounds. Dilutions analyzed as a result of matrix interference elevated the quantitation limit by the dilution factor.

Compound results were frequently reported below CRQLs. Detected results reported below the CRQL were qualified as estimated (LJ). Quantitation limits may be greater than project-required quantitation limits due to dilutions required when compound responses exceed calibration ranges and when moisture content is high.

For sediment samples, pesticide and PCB compounds were qualified with a cautionary flag (W) to indicate that the method of analysis (soil method) might be ineffective for analyzing samples with moisture contents in excess of 70 percent. While the detected compounds are confirmed with mass spectral data, the values are estimates since the analytical method is not optimal for the matrix.

Data quality for the maxima with T qualifiers and the magnitude of the maxima relative to the detection limit indicates these data are not convincing evidence that the analyte in question was actually detected at the maximum concentration reported.

Field Duplicates – Fourteen pair of field duplicates were analyzed for soil samples, and one pair was analyzed for sediments samples. The precision of detected results was calculated and evaluated against the QC criterion. Positive results for endrin ketone and 4,4'-DDT were qualified as estimated (J) because of inconsistencies between duplicate results. Inconsistencies noted with pesticide/PCB results might indicate heterogeneity of matrix and uneven distribution of contaminants in matrix.

2.4 CLP TOTAL METALS AND MOLYBDENUM

CLP Target Analyte List (TAL) metals and molybdenum analyses were performed according to the CLP analytical protocol “EPA Contract Laboratory Program Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration (ILM04.1)” and “EPA Contract Laboratory Program Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration (ILM05.2)” (EPA 2000a and 2001), by EPA CLP laboratories. CLP total metals analyses were conducted on the following quantities and types of field and QC samples:

- 139 soil samples, including 45 sieved and 94 unsieved (plus 22 sets of field duplicates and 21 sets of matrix spikes)
- 35 temporary well samples (plus 4 sets of field duplicates and 3 sets of matrix spikes)
- 20 monitoring well samples (plus 2 sets of field duplicates and 2 sets of matrix spikes)
- 1 surface water sample analyzed for both total and dissolved metals (plus 1 set of field duplicates and 1 set of matrix spikes)
- 8 sediment samples (plus 1 set of field duplicates and 1 set of matrix spikes)
- 25 equipment rinsates

In order to achieve DQOs for this project, the QAPP (Tetra Tech 2003b) required that reporting limits for ground water and surface water media be lower than the standard CRQLs achievable by ICP/atomic emission spectroscopy. As such, the newest CLP protocol (ILM05.2) was requested for analysis of metals by ICP/mass spectrometry. In addition, a flexibility clause was implemented to set the project-required reporting limits as contractual limits. By exercising these variations on the basic protocol (ILM05.2), data for five analytes were not reported: calcium, iron, magnesium, potassium, and sodium. Because of the sensitivity of the ICP/mass spectrometry, mineral-related elements abundantly found in ground water and surface water samples are not as accurately measured and often require numerous serial dilutions that further enhance uncertainty of results. To this end, EPA does not require CLP laboratories to report these five analytes when conducting ICP/mass spectrometry analyses. These data are not available to support the risk assessment for ground water and surface water media.

Data validation findings are summarized in the following paragraphs.

Holding Times and Preservation – The 180-day analytical holding time for metals (and 28-day holding time for mercury) was met for all sample analyses.

Instrument Calibration and Calibration Checks – Initial and continuing calibrations were performed at the proper concentrations and frequencies. All technical criteria were met, and no data were qualified based on calibration criteria.

Instrument detection limits are checked using a contract-required detection limit (CRDL) standard check sample. The CRDL standard results indicated that the instrument was unable to meet the CRDL for several analytes: mercury, selenium, and thallium. As such, results for these analytes were qualified as estimated and biased low (UJv or Jv). However, in sample TWD4-01, the mercury SQL was rejected (UR) because the detection limit was unachievable. In addition, positive results for arsenic, cadmium, and lead at or near the detection limit were qualified as estimated and biased high (J^) because of high recoveries in the CRDL standard.

Aluminum results were qualified as estimated (J) because their response exceeded the upper calibration range of the instrument. The laboratory did not analyze a dilution to bring the response into the calibration range in this case.

Method Blanks – Method and calibration blanks contained trace levels of numerous metals. However, the reviewer indicated that the source of the positive results was most probably instrument noise or drift, instead of actual laboratory contamination. However, sample results for the following metals were qualified as undetected (U) or undetected at an elevated quantitation limit (UC):

Aluminum	Antimony	Arsenic	Beryllium	Cadmium	Calcium
Cobalt	Iron	Lead	Magnesium	Manganese	Mercury
Molybdenum	Nickel	Silver	Sodium	Selenium	Thallium
Vanadium					

In addition, results for arsenic, cadmium, and selenium in soil samples were qualified as estimated and biased high (J^) when positive sample results were greater than 5 times the blank value. Some arsenic and mercury results were qualified as estimated and biased low (Jv) when raw numerical values for blanks were negative.

Equipment Rinsate Blanks – Twenty-five equipment rinsate blanks were collected and analyzed for metals to measure the effectiveness of sampling equipment decontamination procedures. The following metals were detected in equipment blanks:

Aluminum	Antimony	Arsenic	Barium	Beryllium	Calcium
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Chromium	Cobalt	Copper	Iron	Lead	Magnesium
Manganese	Mercury	Nickel	Potassium	Selenium	Silver
Sodium	Vanadium	Zinc			

All detections are less than the CRQL, so no significant impact on sample results is expected. In addition, many of these metals were already qualified as undetected based on method and calibration blanks. Since most of these analytes are also detected in method blanks, no real conclusion about sampling equipment decontamination can be drawn. The source of equipment rinsate contamination cannot be determined.

Pre-Digestion Matrix Spikes – Appropriate analytes were spiked into samples, and MS percent recoveries were evaluated. Results for aluminum, antimony, arsenic, cobalt, copper, manganese, selenium, silver, and vanadium were qualified as estimated and biased low (UJv or Jv) in samples due to low recoveries in the MS. In addition, cobalt, lead, manganese results in some samples were qualified as estimated and biased high (J[^]) due to high MS recoveries.

Laboratory Duplicates – Laboratory duplicates (also know as matrix duplicates [MD]) were analyzed according to the CLP protocol. RPDs were generally within QC limits. However, positive results for aluminum, calcium, chromium, copper, iron, manganese, and zinc were qualified as estimated (J) due to high duplicate RPDs (poor MD duplicate precision).

ICP Quality Control – Serial dilutions and interference check samples required by the CLP protocol were analyzed at the proper frequency and generally met QC criteria. However, manganese, potassium, copper, and nickel results were qualified as estimated and biased high (J[^]) because of the potential for matrix interference. Barium, cadmium, cobalt, copper, nickel, sodium, vanadium, and zinc results were qualified as estimated and biased low (UJv or Jv) because of potential for matrix interference.

The coefficient of variance is also reviewed to ensure instrument precision in measurements. Results for arsenic, lead, selenium, and thallium were qualified as estimated (UJ or J) because replicate measurements of the same sample were inconsistent.

Some samples were analyzed by ICP/MS, which have internal standard requirements, in addition to the other criteria for ICP. Results for aluminum and beryllium in a few samples were qualified as estimated (UJ or J) because of poor internal standard response. In four samples, all analytes were qualified as estimated (UJ or J) because of poor internal standard responses.

Laboratory Control Sample – LCSs were prepared and analyzed at the proper frequency and concentrations. All LCS recoveries were within acceptable QC limits, and no data were qualified on this basis.

Sample Verification – Analyte concentrations and identifications were reviewed, and all sample results were correctly reported. However, the raw data thallium results for several samples were negative numbers, which might indicate suppression of the analytical signal. As such, results for thallium in these samples were qualified as estimated and biased low (UJv or Jv). Quantitation limits may be greater than project-required quantitation limits due to dilutions required when compound responses exceed calibration ranges and when moisture content is high.

All sample results that were greater than the instrument detection limit, but less than the CRDL, were qualified as estimated (L).

Performance Evaluation Samples – Performance evaluation (PE) samples were analyzed and reviewed during validation. Positive antimony, barium, calcium, and zinc results were qualified as estimated and biased high (J[^]) in some samples due to high results in the PE sample. Silver results were qualified as estimated and biased low (UJv or Jv) due to low results in the PE sample.

Field Duplicates – Thirty pair of field duplicates were analyzed for samples (including all matrices sampled). The precision of detected results was calculated and evaluated against the QC criterion. Positive results for chromium, copper, iron, lead, manganese, nickel, and zinc were qualified as estimated (J) because of inconsistencies between duplicate results. Inconsistencies noted with metals results might indicate heterogeneity of matrix and uneven distribution of contaminants in matrix.

2.5 NON-CLP ANIONS AND TOTAL METALS

EPA Region 6 Laboratory in Houston, Texas performed non-CLP analysis of anions and total metals. Anions (including bromide, chloride, fluoride, nitrate, nitrite, and sulfate) were analyzed according to EPA Method 300.0 (EPA 1983), and total metals were analyzed by the following EPA methods:

- ICP metals – Method 200.7 (EPA 1983)
- Graphite furnace metals – Method 200.9 (EPA 1983)
- Mercury – Method 245.1 (EPA 1983)

Non-CLP anions and total metals analyses were conducted on the following quantities and types of field and QC samples:

- 102 soil samples for molybdenum only (plus 16 sets of field duplicates)
- 35 temporary well samples for molybdenum only (plus 4 sets of field duplicates)
- 27 equipment rinsate samples for molybdenum only 1 equipment rinsate for anions
- 24 monitoring well samples for anions (plus 2 sets of field duplicates) and 4 monitoring well samples for TAL metals (plus 1 set of field duplicates)
- 1 equipment rinsate for anions

EPA Region 6 laboratory data did not require validation as specified in the project-specific QAPP (Tetra Tech 2003b). As such, the reports contained a cover letter with general information regarding data quality. The issues reported in cover letters are summarized in the following paragraphs. The laboratory rejected no data, so all were usable as reported.

Due to an oversight, nitrate and nitrite analyses for four monitoring well ground water samples were conducted past the recommended holding time. Nitrate and nitrite results for samples MW03-01, MW08-01, MW17-01, and MW18-01 should be considered estimated and possibly biased low.

According to the narrative provided by the EPA Region 6 laboratory, MS recoveries were high for aluminum and molybdenum and low for barium. As such, results for aluminum and molybdenum may be biased high, and results for barium may be biased low. In addition, both the MS and LCS recoveries for silver were low. Silver data should not be biased; however, Tetra Tech chemists recommend that silver data be considered biased low based on MS and LCS recoveries.

2.6 NON-CLP WASTE CHARACTERIZATION

Waste samples were collected during the on-site investigation. Tetra Tech contractor laboratories conducted waste analysis and characterization according to the following test methods:

- Total metals (including mercury and molybdenum) – Method 6010B, 6020, and 7470 (EPA 1996)
- Toxicity characteristic leaching procedure (TCLP) metals – Method 1311 followed by Method 6010B, 6020, and 7470 (EPA 1996)

- Total VOC – Method 8260B (EPA 1996)
- TCLP VOC – Method 1311 followed by Method 8260B (EPA 1996)
- Total SVOC – Method 8270C (EPA 1996)
- TCLP SVOC – Method 1311 followed by Method 8270C (EPA 1996)
- Total pesticides/PCBs – Methods 8081A and 8082 (EPA 1996)
- Total petroleum hydrocarbons (TPH) – Method TX 1005 (Texas Natural Resource Conservation Commission 2001)
- Reactivity, corrosivity, and ignitability (RCI) – SW-846 Chapter 7 (EPA 1996)

Table 4 indicates the sample identification numbers, matrix, and analyses conducted. According to the project-specific QAPP (Tetra Tech 2003b), Tetra Tech was responsible for conducting data validation on all non-CLP contractor laboratories. Tetra Tech conducted the validation according to validation guidelines listed in Section 2.0. DVRs are provided in Appendix A. The following paragraphs describe data usability and qualifiers applied data.

Suspected Catalyst Material and Foundry Sand and Slag – These samples include WC-01 through WC-08 and were analyzed by e-Lab for a short list of total metals and TCLP metals according to Table 4. QC criteria for these analyses were generally met with the following exceptions:

- A total molybdenum result was qualified as undetected (U) due to blank contamination.

TABLE 4
WASTE SAMPLE ANALYSIS SCHEME

Sample ID	Waste Type	Analysis
WC-01	Suspected catalyst material	Total metals ¹
WC-02	Suspected catalyst material	Total metals ¹
WC-03	Suspected catalyst material	Total metals ¹ and TCLP metals ¹
WC-04	Suspected catalyst material	Total metals ¹
WC-05	Suspected catalyst material	Total metals ¹
WC-06	Loose foundry sand	Total metals ¹
WC-07	Foundry sand from casting	Total metals ¹
WC-08	Suspected foundry slag	Total metals ¹ and TCLP metals ¹
Waste-01	Liquid waste	Total metals ² , SVOC, VOC, PCB
Waste-02	Liquid waste	Total metals ² , SVOC, VOC, PCB
Waste-03	Liquid waste	Total metals ² , SVOC, VOC, PCB
Waste-04	Solid waste material	Total metals ² , SVOC, VOC, PCB, pesticides
Waste-05	Liquid waste	Total metals ² , SVOC, VOC, PCB
Waste-06	Liquid waste	Total metals ² , SVOC, VOC, PCB
Waste-07	Liquid waste	Total metals ² , SVOC, VOC, PCB
Waste-08	Liquid waste	Total metals ² , SVOC, VOC, PCB
Waste-09	Solid waste material	VOC, SVOC, TX1005, pesticides/PCB, total metals ² , TCLP metals ³ , RCI
Waste-09A	Liquid waste	VOC, SVOC, TX1005, pesticides/PCB, total metals ² , RCI
Baghouse #1	Baghouse filter medium	Total metals ² , TCLP metals ³
Baghouse #2	Baghouse filter medium	Total metals ² , TCLP metals ³
Baghouse #3	Baghouse filter medium	Total metals ² , TCLP metals ³
Baghouse #4	Baghouse filter medium	Total metals ² , TCLP metals ³
Baghouse #5	Baghouse filter medium	Total metals ² , TCLP metals ³
IDW-01	Investigation-derived waste	TCLP VOC, TCLP SVOC, TCLP metals ³ , RCI
Portland cement	Cement sample	Total metals ²

Notes:

- 1 Metals include arsenic, barium, cadmium, chromium, cobalt, copper, lead, mercury, molybdenum, nickel, selenium, and silver
- 2 Metals include the target analyte list metals.
- 3 Metals include arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver

IDW Investigation-derived waste	PCB Polychlorinated biphenyl
RCI Reactivity, corrosivity, ignitability	SVOC Semivolatile organic compound
TCLP Toxicity Characteristic Leaching Procedure	VOC Volatile organic compound

- Total chromium, copper, molybdenum, and nickel results for samples were qualified as estimated (J) and possibly biased high due to high MS recoveries.
- TCLP mercury results were qualified as estimated (UJ or J) and possibly biased low due to holding time exceedance.
- TCLP cadmium, cobalt, copper, lead, and molybdenum results were qualified as undetected (U) due to blank contamination.
- TCLP molybdenum results were qualified as estimated (J) and possibly biased high due to high MS recoveries.
- All results that were greater than the method detection limit, but less than the reporting limit, were qualified as estimated (J).

Liquid and Solid Waste Material – These samples include Waste-01 through Waste-09A and were analyzed by Accutest according to the listing in Table 4. QC criteria for each of these analyses were generally met with the following exceptions:

Volatile aromatic compounds were qualified as estimated (UJ or J) due to high pH of samples upon receipt to laboratory (greater than pH 2).

Common laboratory contaminants, including acetone, methylene chloride, and bis(2-ethylhexyl)phthalate results were qualified as undetected (U) due to blank contamination.

- Dichlorofluoromethane, β -BHC, and γ -BHC results were qualified as estimated (UJ) due to low LCS and MS recoveries.
- Endosulfan sulfate results were qualified as estimated (J) and possibly biased high due to high MS recoveries.
- Endosulfan I, antimony, lead, molybdenum, and potassium results were qualified as estimated (UJ or J) due to low MS recoveries.
- Beryllium results were qualified as undetected (U) due to blank contamination.
- All results that were greater than the method detection limit, but less than the reporting limit, were qualified as estimated (J).

Baghouse Filter Material – These samples include Baghouse #1 through Baghouse #5 and were analyzed by Accutest according to the listing in Table 4. QC criteria for each of these analyses were generally met with the following exceptions:

- Antimony, lead, molybdenum, and potassium results were qualified as estimated (UJ or J) due to low MS recoveries.
- All results that were greater than the method detection limit, but less than the reporting limit were qualified as estimated (J).

IDW and Portland Cement Samples – These samples include IDW-01 and Portland Cement and were analyzed by Accutest according to the listing in Table 4. QC criteria for each of these analyses were generally met with the following exceptions:

- Antimony, lead, molybdenum, and potassium results were qualified as estimated (UJ or J) due to low MS recoveries.
- All results that were greater than the method detection limit, but less than the reporting limit, were qualified as estimated (J).

2.7 NON-CLP REMEDY SAMPLES

Remedy samples were collected during the on-site investigation to obtain general chemistry, mobility, and geotechnical data for the feasibility study. A total of 18 soil samples (plus 2 sets of field duplicates) were analyzed for the remedy suite. Tetra Tech contractor laboratories conducted remedy suite analysis according to the following test methods

- Total metals (including mercury) – Method 6010B and 7471A (EPA 1996)
- TCLP metals – Method 1311 followed by Method 6010B and 7470A (EPA 1996)
- Anions (including bromide, chloride, fluoride, nitrate, nitrite, and sulfate) – Method 9056 (EPA 1996)
- Total phenols – Method 365.2 modified for soil samples (EPA 1983)
- Sulfide – Method 9031 (EPA 1996)
- Sulfite – Method 377.1 (EPA 1983)

According to the project-specific QAPP (Tetra Tech 2003b), Tetra Tech was responsible for conducting data validation on all non-CLP contractor laboratories. Tetra Tech conducted the validation according to validation guidelines listed in Section 2.0. DVRs are presented in Appendix A. The following paragraphs describe data usability and qualifiers applied data.

Total antimony results were qualified as estimated (UJ/J) due to MS recoveries. The MS recovery was greater than the QC limits, and the MSD recovery was less than the QC limits, so no bias could be determined. In addition, total calcium, copper, and zinc results (positive values only) were qualified as estimated (J) due to high MS and MSD recoveries. These results may be biased high.

Total aluminum, arsenic, calcium, chromium, cobalt, copper, lead, magnesium, manganese, nickel, and potassium results (positive values only) were qualified as estimated (J) due to poor duplicate precision. TCLP chromium data (positive values only) were also qualified as estimated (J) due to poor duplicate precision. Poor duplicate precision may indicate poor laboratory precision or the heterogeneity of the matrix.

Total cadmium and potassium results (positive values only) were qualified as estimated (J) because the ICP serial dilution results indicate the potential for matrix interference.

TCLP chromium, arsenic, and silver results were qualified as estimated (UJ or J) due to low MS and MSD recoveries. The results may be biased low.

All results that were greater than the method detection limit, but less than the reporting limit, were qualified as estimated (J).

2.8 FIELD-BASED METALS ANALYSES

In order to have real-time analytical results for critical parameters at the site and to reduce the total analytical expenditures, soil samples were prepared and analyzed for total metals (including arsenic, chromium, cobalt, copper, iron, lead, manganese, mercury, molybdenum, nickel, and zinc) using field-based (XRF) instrumentation (EPA 1998). A total of 942 soil samples (plus 63 sets of field duplicates) were prepared and analyzed using this technique. Field QC samples included field duplicates and a calibration verification sample. All calibration verification sample results met QC criteria. Variation in results for field duplicates were generally acceptable, and did not affect overall data quality.

At 80 locations (8.5 percent of the total number of sample locations), soil samples were split for confirmatory purposes and analyzed for total metals at an off-site laboratory (CLP laboratory, EPA Region 6 laboratory, or Tetra Tech contractor laboratory). For the most part, field-based XRF analyses conducted at the MDI site are rated as screening level data. A comparison of XRF data to definitive data (obtained for this project at off-site laboratories) was conducted in an attempt to elevate the data quality to

a level suitable for use in determining the nature and extent, as well as risk assessment. Specifically, it is well known that there is a strong correlation between XRF and laboratory results for lead. Tetra Tech wanted to capitalize on this correlation to collect data more cost-effectively, while also determining if similar correlations existed for other metals at the site. As such, statistical evaluation of the two data sets for the 80 collocated sites was performed. The statistical analyses, plots, and conclusions are presented in Section 3.0.

3.0 EVALUATION OF LABORATORY DATA VERSUS XRF DATA

At 80 sample locations, soil samples were collected, homogenized, and split into two equal aliquots, both of which were analyzed using field-based XRF and off-site fixed laboratory resources. Each dataset was reviewed for accuracy, then used for comparative purposes. The purpose was to evaluate the effectiveness of the field-based XRF data for making decisions regarding nature and extent, and the evaluation of risks to human health and the environment. The following sections discuss the statistical tests conducted on each dataset and the conclusions of the evaluation.

3.1 STATISTICAL TESTS AND PLOTS

Initially, basic descriptive statistics were calculated on each set of analytical data (laboratory and XRF) from the 80 collocated samples. These statistics include: (1) sample size of total and detected results; (2) detection frequency; and (3) minimum, maximum, and mean concentrations for each dataset. These statistical analyses were conducted on both detected and non-detected data, where one half the reporting limit was used for non-detect results. These basic descriptive statistics are summarized in Table 5 and depicted in as box plots and quantile tables on Figures 1A through 1F. Figure 2 provides the key for interpreting box plots.

Comparisons between the two datasets were conducted using two statistical techniques: (1) linear regression analysis, and (2) the nonparametric, two-population Wilcoxon Rank Sum (WRS) test. As stated previously, non-detected results were valued at one half the reporting limit for comparison purposes. Results of the linear regression analysis of the 80 collocated laboratory and XRF raw metals concentrations are plotted on Figures 3A through 3C. A detailed linear regression analysis was performed for lead using all raw data and natural-log transformed data. In addition, another set of linear regression analyses using raw data and natural-log transformed data were conducted excluding the single outlier. Results of these four analyses are plotted on Figure 4. For lead only, a WRS comparison of the data was conducted using (1) laboratory data, (2) adjusted XRF data, and (3) combined laboratory and

TABLE 5

SUMMARY STATISTICS COMPARING LABORATORY AND XRF DATA

Chemical	Laboratory Data						XRF Data					
	Sample Size		Detection Frequency (Percent)	Concentration (mg/kg) ¹			Sample Size		Detection Frequency (Percent)	Concentration (mg/kg) ¹		
	Detected	Total		Minimum	Median	Maximum	Detected	Total		Minimum	Median	Maximum
Arsenic	74	80	92	0.42	4.80	56.90	0	80	0	5.03	13.13	84.38
Chromium	80	80	100	1.50	57.15	482.50	9	80	11	78.75	255.94	4,348.80
Cobalt	80	80	100	1.10	5.58	306.00	25	80	31	120.94	430.31	7,142.40
Copper	80	80	100	3.30	51.30	478.00	17	80	21	15.61	48.09	1,440.00
Iron	80	80	100	926.00	20,500.00	91,300.00	80	80	100	5,318.40	26,243.20	195,430.40
Lead	80	80	100	3.70	46.30	2,050.00	44	80	55	6.78	48.95	2,560.00
Manganese	80	80	100	32.10	440.50	12,200.00	54	80	68	194.06	2,049.60	20,441.60
Mercury	26	80	32	0.01	0.03	0.94	0	80	0	2.67	6.95	48.43
Molybdenum	50	63	79	0.63	16.30	209.00	24	63	38	2.14	16.99	154.10
Nickel	80	80	100	2.70	49.65	489.00	4	80	5	27.90	87.19	1,788.80
Zinc	80	80	100	9.10	119.00	2,910.00	52	80	65	9.11	141.73	4,819.20

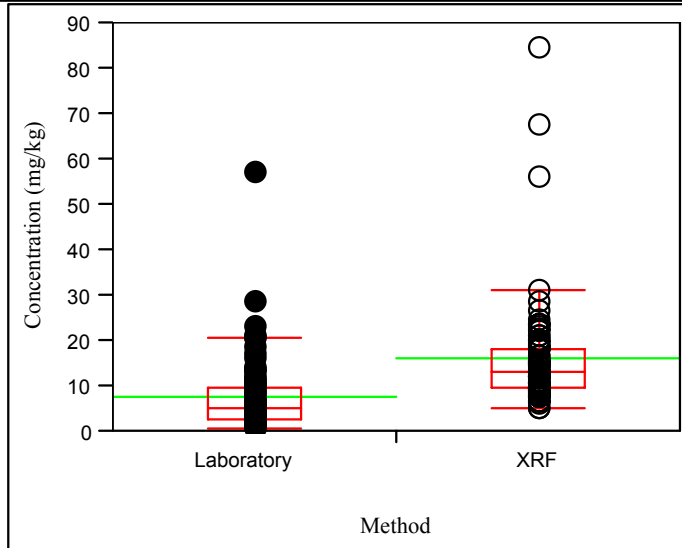
Notes:

- 1 Analysis was based on the detected and non-detected data combined. One-half of the reporting limit was substituted for all nondetect values.
- mg/kg Micrograms per kilograms

FIGURE 1
COMPARISON OF LABORATORY AND XRF RESULTS USING BOX PLOTS AND
QUANTILE TABLES

Metal=Arsenic

Oneway Analysis of Concentration (mg/kg) By Method

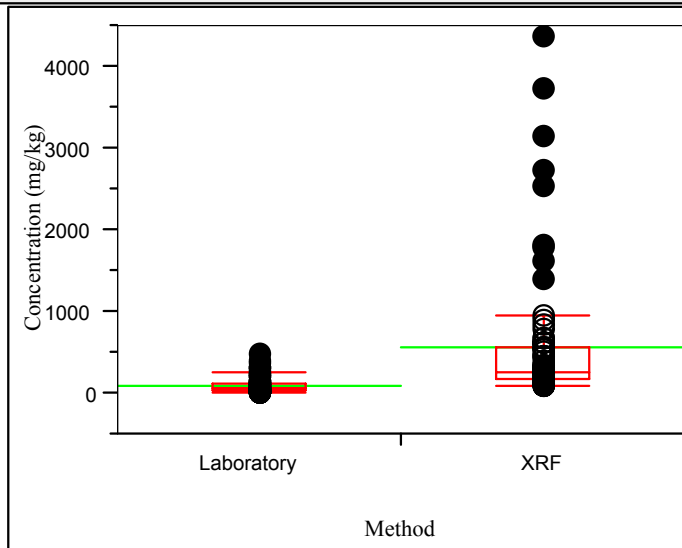


Quantiles

Level	Minimum	10%	25%	Median	75%	90%	Maximum
Laboratory	0.415	1.26	2.5	4.8	9.725	16.97	56.9
XRF	5.034375	7.666875	9.407813	13.13438	18.19687	23.70375	84.375

Metal=Chromium

Oneway Analysis of Concentration (mg/kg) By Method



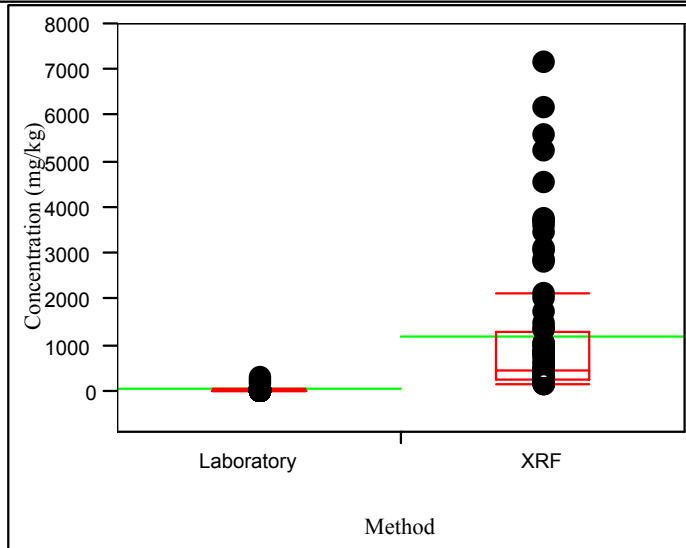
Quantiles

Level	Minimum	10%	25%	Median	75%	90%	Maximum
Laboratory	1.5	4.06	16.475	57.15	117.9625	246	482.5
XRF	78.75	120.9375	168.75	255.9375	546.9375	1580	4348.8

FIGURE 1 (CONTINUED)
COMPARISON OF LABORATORY AND XRF RESULTS USING BOX PLOTS AND
QUANTILE TABLES

Metal=Cobalt

Oneway Analysis of Concentration (mg/kg) By Method

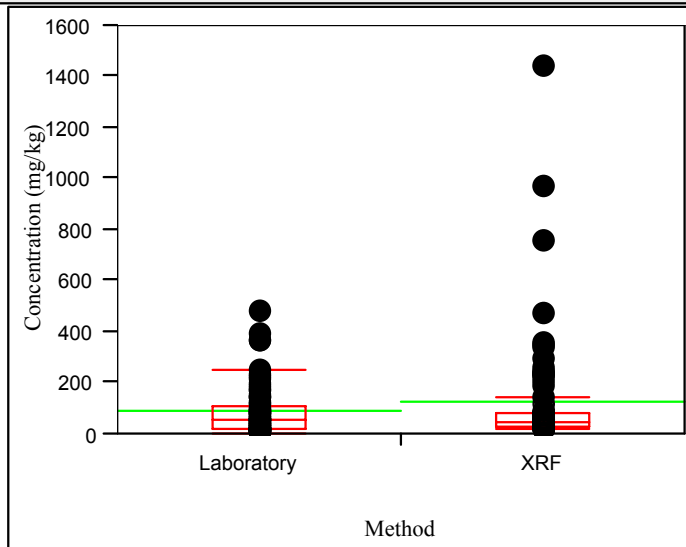


Quantiles

Level	Minimum	10%	25%	Median	75%	90%	Maximum
Laboratory	1.1	1.71	3.25	5.575	10.175	27.81	306
XRF	120.9375	177.4688	254.5312	430.3125	1252.2	3688	7142.4

Metal=Copper

Oneway Analysis of Concentration (mg/kg) By Method



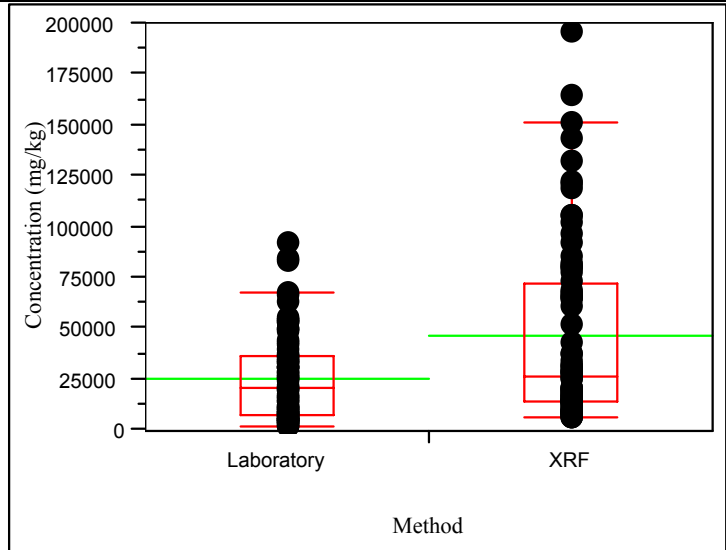
Quantiles

Level	Minimum	10%	25%	Median	75%	90%	Maximum
Laboratory	3.3	9.76	20.325	51.3	111.075	210.9	478
XRF	15.60938	24.77812	29.65781	48.09375	84.375	292.56	1440

FIGURE 1 (CONTINUED)
COMPARISON OF LABORATORY AND XRF RESULTS USING BOX PLOTS AND QUANTILE TABLES

Metal=Iron

Oneway Analysis of Concentration (mg/kg) By Method

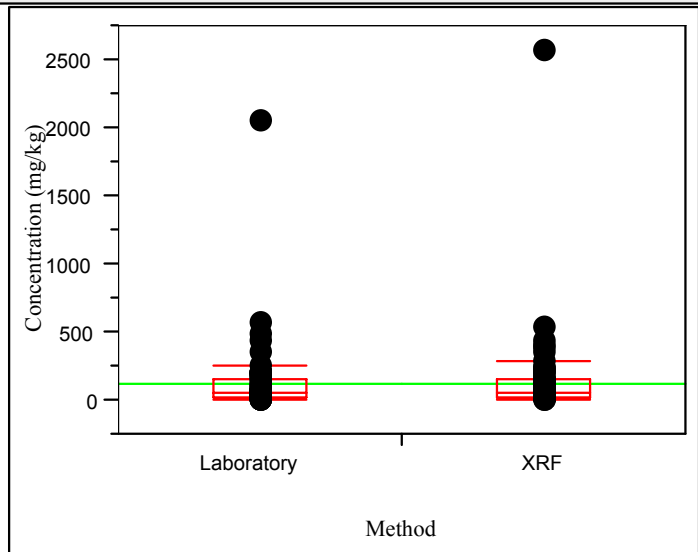


Quantiles

Level	Minimum	10%	25%	Median	75%	90%	Maximum
Laboratory	926	3851	7267.5	20500	36100	54140	91300
XRF	5318.4	8132.48	13491.2	26243.2	71200	117585.9	195430.4

Metal=Lead

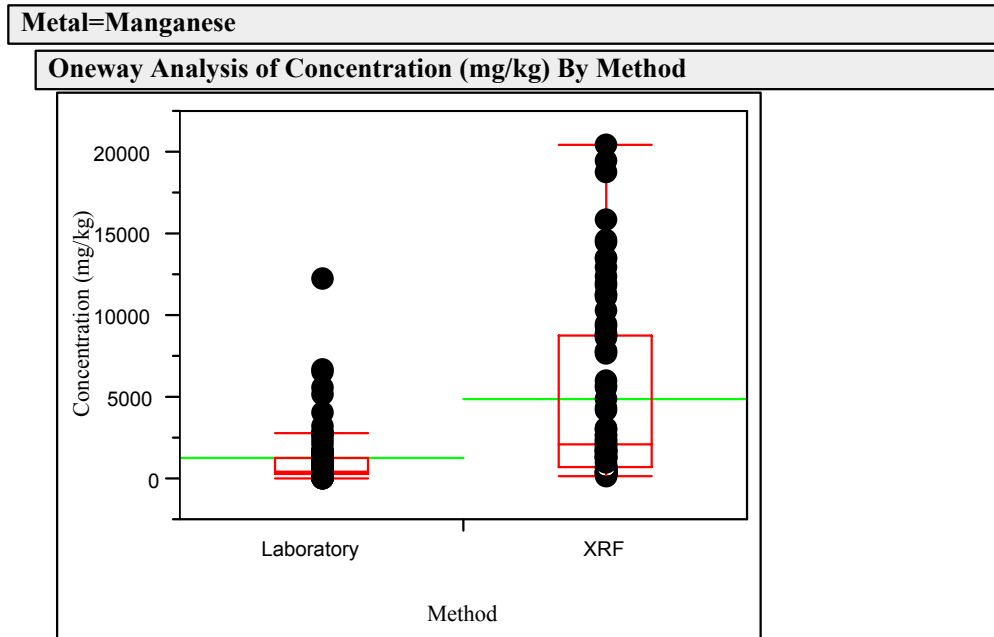
Oneway Analysis of Concentration (mg/kg) By Method



Quantiles

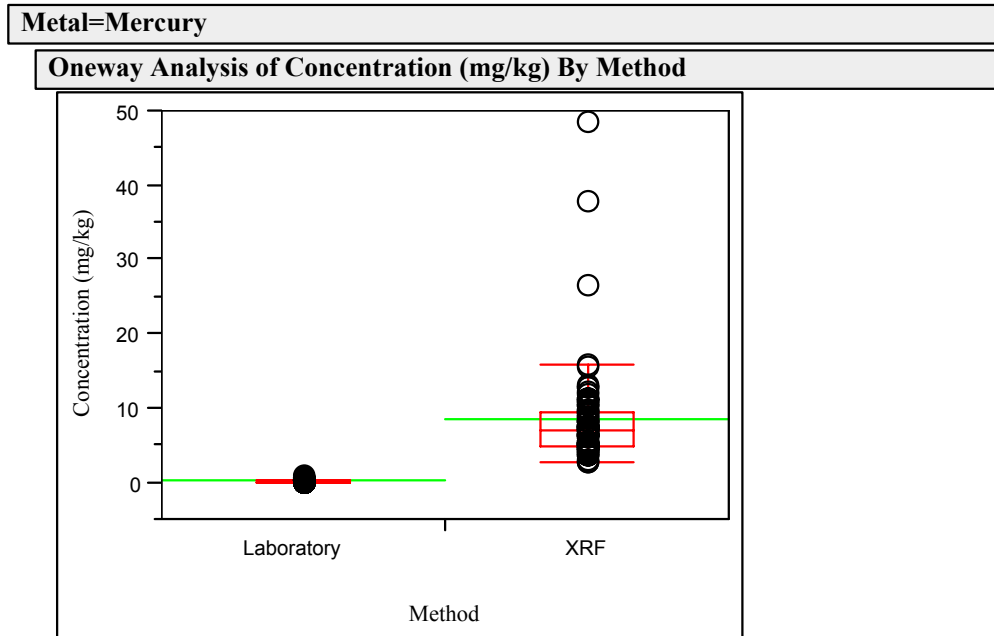
Level	Minimum	10%	25%	Median	75%	90%	Maximum
Laboratory	3.7	9.2	15.9	46.3	142.5	204.7	2050
XRF	6.778125	12.0825	17.35312	48.95	142.3	240.03	2560

FIGURE 1 (CONTINUED)
COMPARISON OF LABORATORY AND XRF RESULTS USING BOX PLOTS AND
QUANTILE TABLES



Quantiles

Level	Minimum	10%	25%	Median	75%	90%	Maximum
Laboratory	32.1	117.8	225	440.5	1266.25	2813.5	12200
XRF	194.0625	345.375	675	2049.6	8712	13431.68	20441.6



Quantiles

Level	Minimum	10%	25%	Median	75%	90%	Maximum
Laboratory	0.0125	0.02	0.025	0.025	0.08	0.157	0.94
XRF	2.671875	3.945937	4.907813	6.946875	9.45	12.54375	48.43125

FIGURE 1 (CONTINUED)
COMPARISON OF LABORATORY AND XRF RESULTS USING BOX PLOTS AND
QUANTILE TABLES

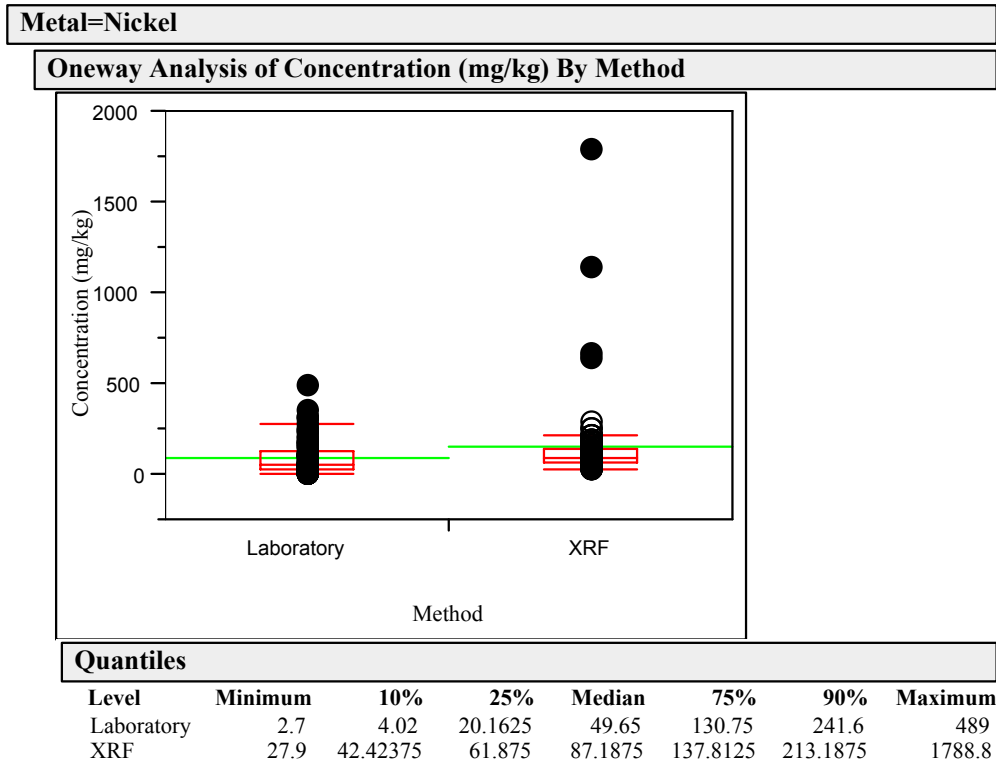
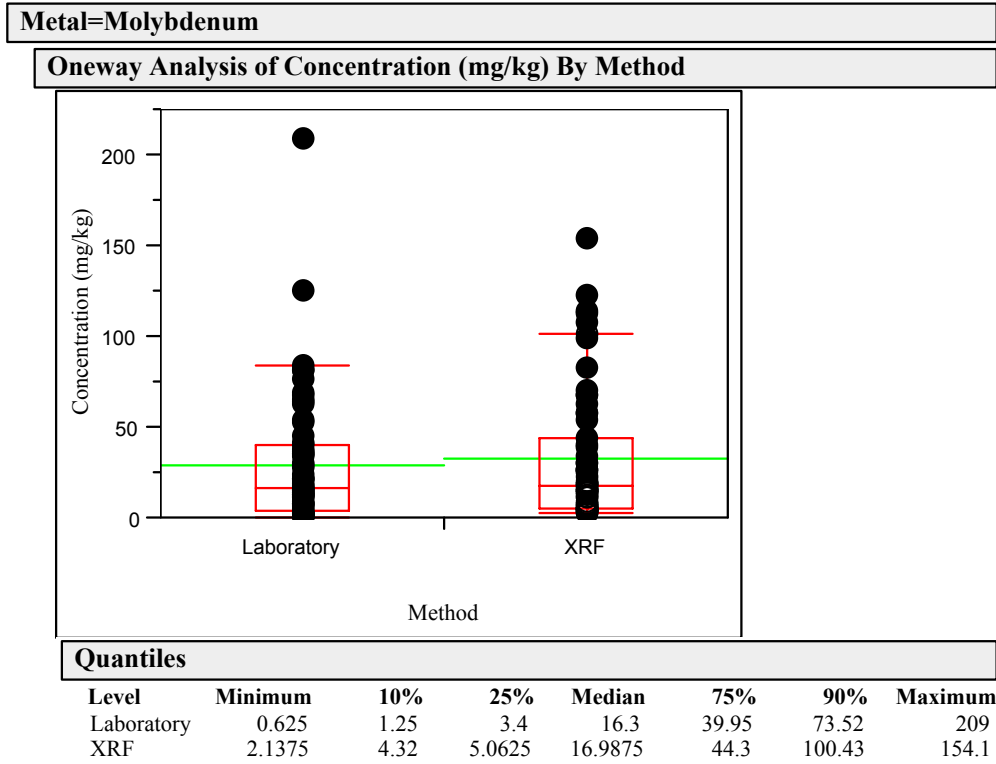
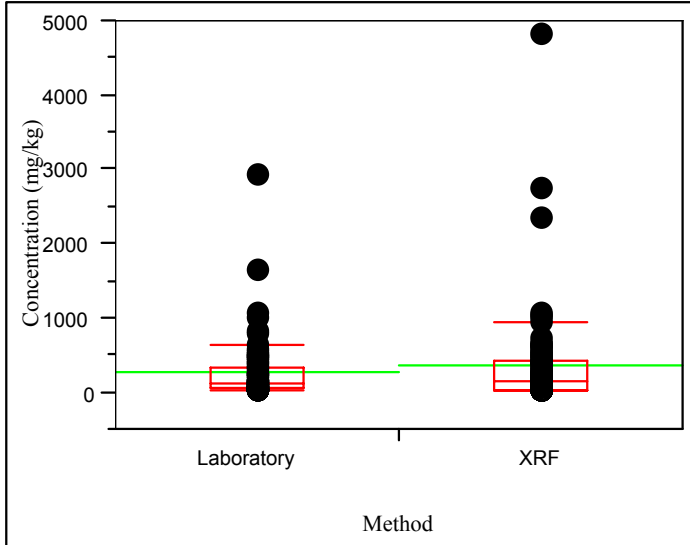


FIGURE 1 (CONTINUED)
COMPARISON OF LABORATORY AND XRF RESULTS USING BOX PLOTS AND
QUANTILE TABLES

Metal=Zinc

Oneway Analysis of Concentration (mg/kg) By Method



Quantiles

Level	Minimum	10%	25%	Median	75%	90%	Maximum
Laboratory	9.1	12.44	37.55	119	336	554.1	2910
XRF	9.1125	15.30562	21.6	141.725	408.85	729.02	4819.2

FIGURE 2

KEY FOR INTERPRETING BOX PLOTS

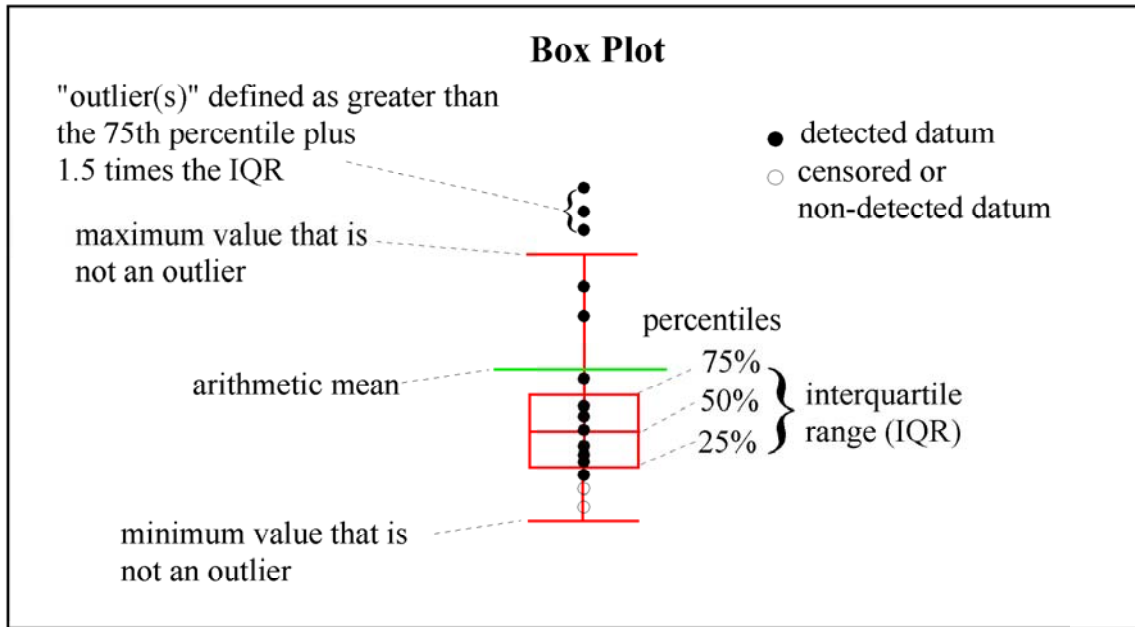
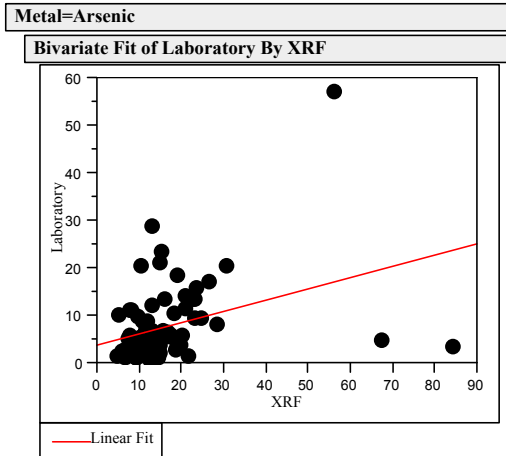


FIGURE 3

RESULTS OF LINEAR REGRESSION ANALYSIS OF COLLOCATED LABORATORY AND XRF DATA



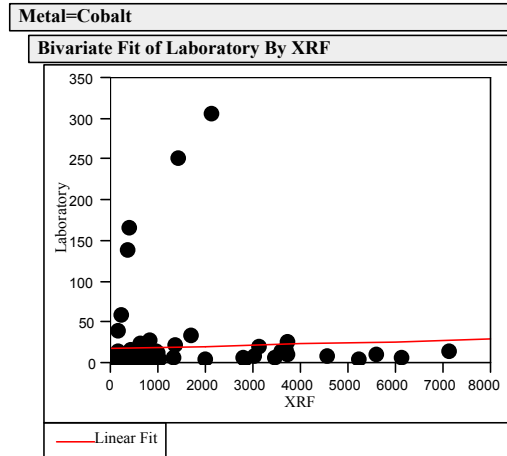
Linear Fit

Laboratory = 3.514278 + 0.2371687 XRF

Summary of Fit	
RSquare	0.125416
RSquare Adj	0.114204
Root Mean Square Error	7.688391
Mean of Response	7.291625
Observations (or Sum Wgts)	80

Analysis of Variance				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	661.1775	661.177	11.1853
Error	78	4610.6854	59.111	Prob > F
C. Total	79	5271.8628		0.0013

Parameter Estimates				
Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	3.514278	1.41934	2.48	0.0155
XRF	0.2371687	0.070914	3.34	0.0013



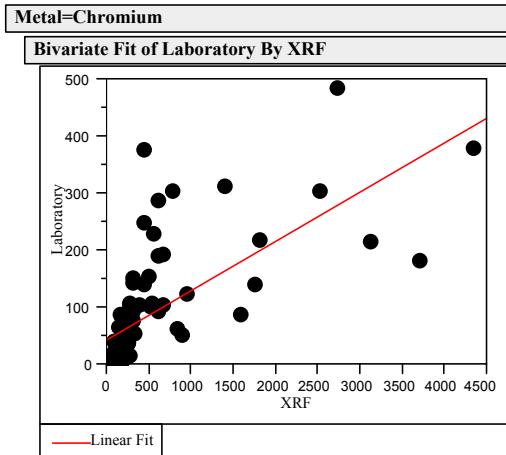
Linear Fit

Laboratory = 17.116617 + 0.0013905 XRF

Summary of Fit	
RSquare	0.001978
RSquare Adj	-0.01082
Root Mean Square Error	48.84346
Mean of Response	18.75313
Observations (or Sum Wgts)	80

Analysis of Variance				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	368.77	368.77	0.1546
Error	78	186083.29	2385.68	Prob > F
C. Total	79	186452.06		0.6953

Parameter Estimates				
Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	17.116617	6.866353	2.49	0.0148
XRF	0.0013905	0.003537	0.39	0.6953



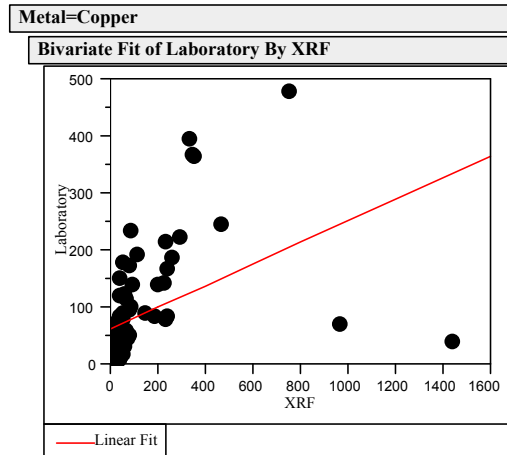
Linear Fit

Laboratory = 42.929792 + 0.085939 XRF

Summary of Fit	
RSquare	0.46606
RSquare Adj	0.459214
Root Mean Square Error	75.24472
Mean of Response	90.545
Observations (or Sum Wgts)	80

Analysis of Variance				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	385474.45	385474	68.0838
Error	78	441617.90	5662	Prob > F
C. Total	79	827092.35		<.0001

Parameter Estimates				
Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	42.929792	10.20159	4.21	<.0001
XRF	0.085939	0.010415	8.25	<.0001



Linear Fit

Laboratory = 61.819481 + 0.1889248 XRF

Summary of Fit	
RSquare	0.181962
RSquare Adj	0.171474
Root Mean Square Error	86.6335
Mean of Response	84.75625
Observations (or Sum Wgts)	80

Analysis of Variance				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	130218.70	130219	17.3501
Error	78	585418.29	7505	Prob > F
C. Total	79	715636.99		<.0001

Parameter Estimates				
Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	61.819481	11.14179	5.55	<.0001
XRF	0.1889248	0.045356	4.17	<.0001

FIGURE 3 (CONTINUED)

RESULTS OF LINEAR REGRESSION ANALYSIS OF COLLOCATED LABORATORY AND XRF DATA

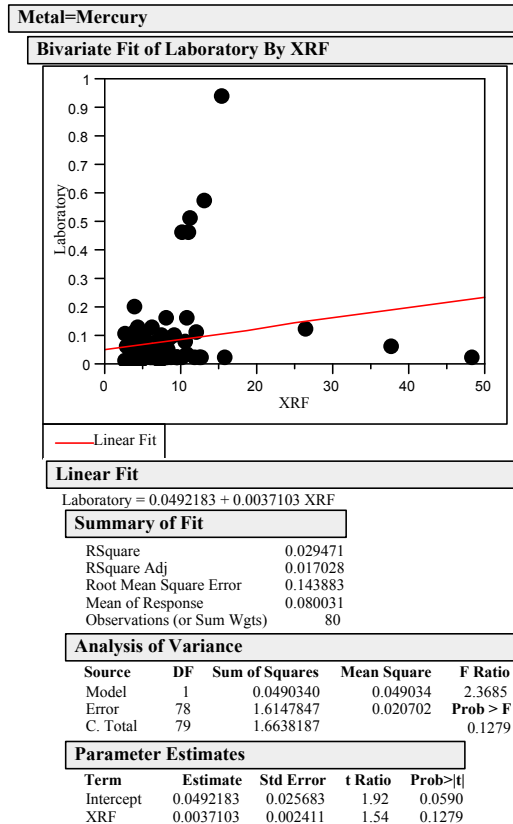
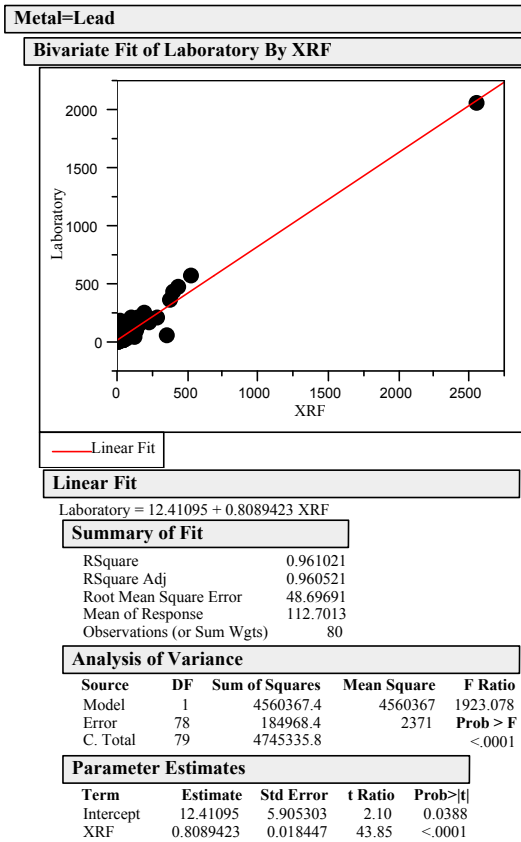
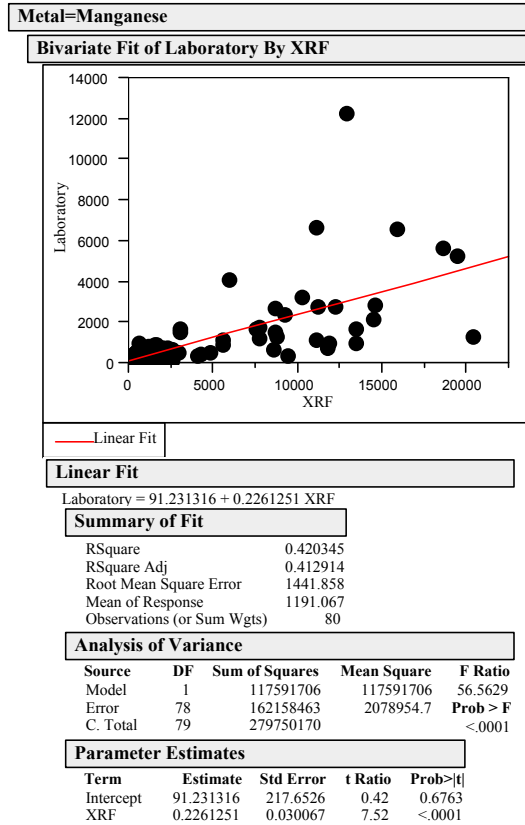
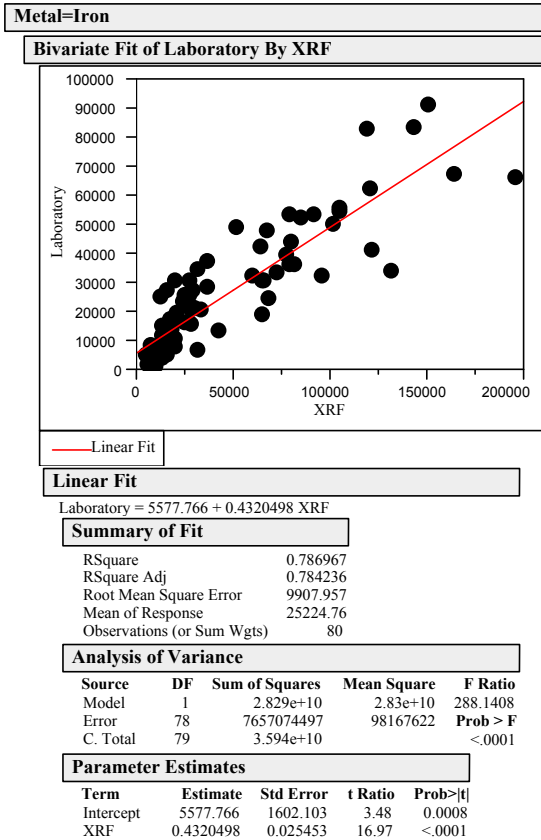


FIGURE 3 (CONTINUED)

RESULTS OF LINEAR REGRESSION ANALYSIS OF COLLOCATED LABORATORY AND XRF DATA

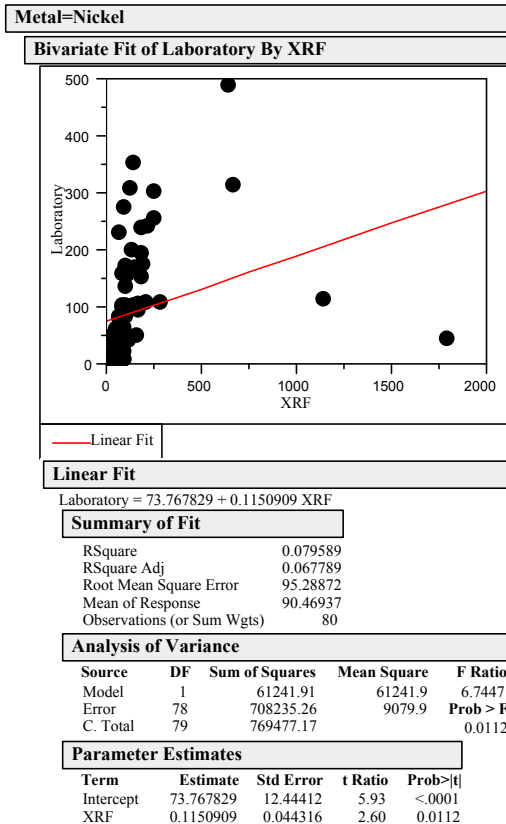
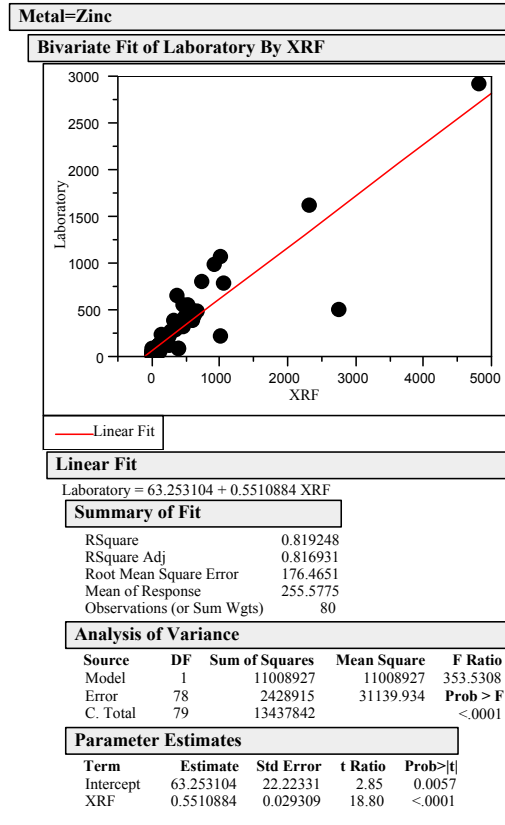
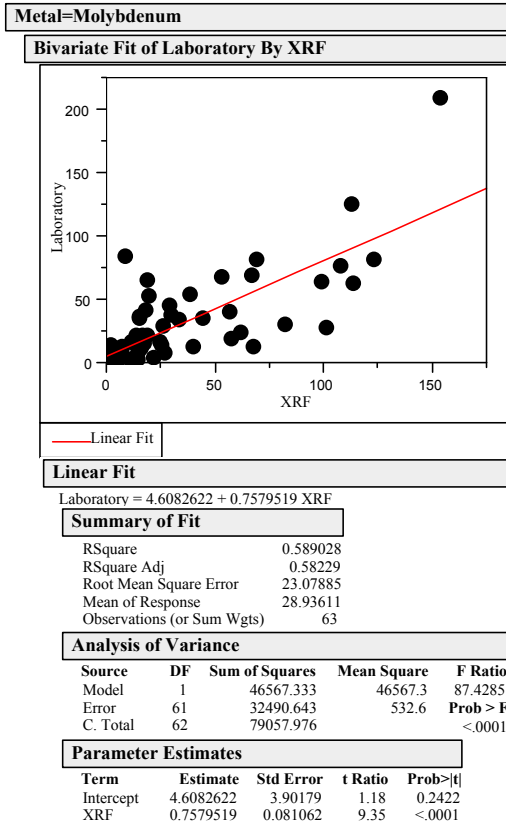
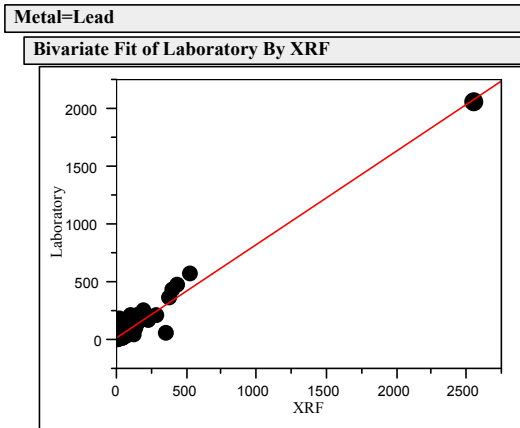


FIGURE 4

RESULTS OF LINEAR REGRESSION ANALYSIS FOR LEAD USING DATA IN ORIGINAL AND NATURAL LOG UNITS AND INCLUDING AND EXCLUDING A SINGLE OUTLIER



Linear Fit

Linear Fit

Laboratory = 12.41095 + 0.8089423 XRF

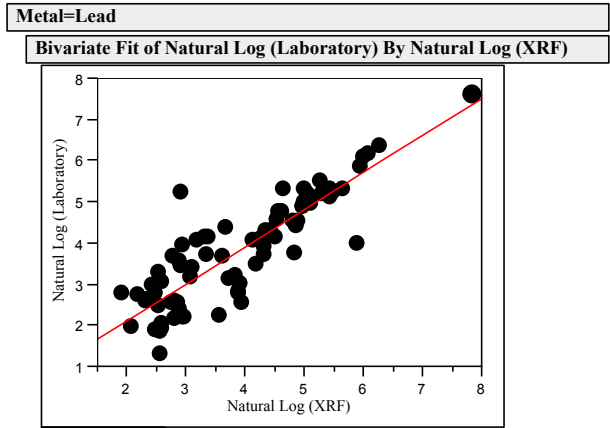
Summary of Fit	
RSquare	0.961021
RSquare Adj	0.960521
Root Mean Square Error	48.69691
Mean of Response	112.7013
Observations (or Sum Wgts)	80

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	4560367.4	4560367	1923.078
Error	78	184968.4	2371	Prob > F
C. Total	79	4745335.8		<.0001

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	12.41095	5.905303	2.10	0.0388
XRF	0.8089423	0.018447	43.85	<.0001



Linear Fit

Linear Fit

Natural Log (Laboratory) = 0.3125012 + 0.8978617 Natural Log (XRF)

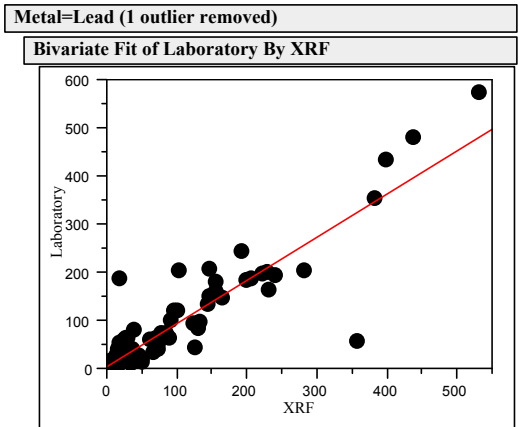
Summary of Fit	
RSquare	0.758383
RSquare Adj	0.755286
Root Mean Square Error	0.635702
Mean of Response	3.844949
Observations (or Sum Wgts)	80

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	98.93833	98.9383	244.8256
Error	78	31.52117	0.4041	Prob > F
C. Total	79	130.45950		<.0001

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0.3125012	0.236684	1.32	0.1906
Natural Log (XRF)	0.8978617	0.057383	15.65	<.0001



Linear Fit

Linear Fit

Laboratory = 4.858234 + 0.8945571 XRF

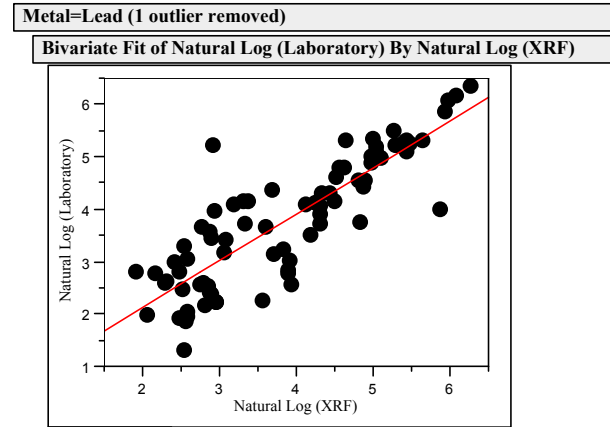
Summary of Fit	
RSquare	0.812839
RSquare Adj	0.810408
Root Mean Square Error	47.91925
Mean of Response	88.17848
Observations (or Sum Wgts)	79

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	767889.83	767890	334.4098
Error	77	176811.57	2296	Prob > F
C. Total	78	944701.40		<.0001

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	4.858234	7.058772	0.69	0.4934
XRF	0.8945571	0.048918	18.29	<.0001



Linear Fit

Linear Fit

Natural Log (Laboratory) = 0.3474445 + 0.8879971 Natural Log (XRF)

Summary of Fit	
RSquare	0.728943
RSquare Adj	0.725423
Root Mean Square Error	0.638979
Mean of Response	3.797092
Observations (or Sum Wgts)	79

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	84.54667	84.5467	207.0732
Error	77	31.43861	0.4083	Prob > F
C. Total	78	115.98528		<.0001

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0.3474445	0.250272	1.39	0.1691
Natural Log (XRF)	0.8879971	0.061709	14.39	<.0001

adjusted XRF data. Adjusted XRF concentrations were derived by using the slope and intercept of the regression analysis to generate laboratory-adjusted concentrations for each collocated XRF measurement. The resultant plot of this comparison is shown on Figure 5. An additional WSR test was conducted to compare all laboratory data and all laboratory-adjusted XRF data for both lead and zinc. These analyses are depicted on Figure 6.

3.2 EVALUATION CONCLUSIONS

A review of the basic descriptive statistical measurements presented in Table 5 indicates that the low detection frequencies (DF) in the XRF dataset are the principal limiting factor in the data usability. Many of the WRS results have to be considered “suspect” because of the low DF in the XRF dataset. For a more accurate interpretation, data from Table 5 should be reviewed along with results from the WSR in Table 6. The XRF data shows greater variability than the fixed lab data, and the distributions of most metals analyzed by XRF are biased higher as compared to the laboratory data.

The linear regression analysis (depicted on Figures 3A through 3C and summarized in Table 6) indicates a poor fit for most metals. Using the correlation coefficient as an indication of linearity (1.000 being perfectly linear), the analysis indicates that lead data has the strongest linear relationship between the variables (a correlation coefficient of 0.961), with zinc having the next best linear relationship (a correlation coefficient of 0.819). The second set of linear regression calculations (using natural-log transformed data and/or eliminating the outlier) was a test to determine whether the distribution or presence of an outlier enhanced the linear relationship between datasets (Figure 4). However, the best correlation coefficient was obtained from the initial linear regression analysis using raw data that included the outlier (see upper-left plot on Figure 4). Accordingly, additional statistical manipulation of the data proved to be unwarranted.

Because of the overall similarity of the XRF and laboratory datasets for lead (from linear regression analysis), a comparison of three types of lead data (laboratory lead data, adjusted XRF data, and combined laboratory and adjusted XRF lead data) was conducted to determine the effect of using laboratory data alone or in combination with XRF lead data. The plot on Figure 5 indicates that if the adjusted XRF data are used in combination with laboratory data for lead, then the upper-range of lead concentrations as well as the estimated variability within the dataset is increased.

Since lead and zinc results have the best linear relationships between datasets, further comparison was conducted. First, the XRF data were adjusted using the slope and intercept of the regression analysis to

FIGURE 5

TWO-POPULATION COMPARISON OF LABORATORY AND ADJUSTED XRF RESULTS FOR LEAD AND ZINC USING THE WILCOXON RANK SUM TEST

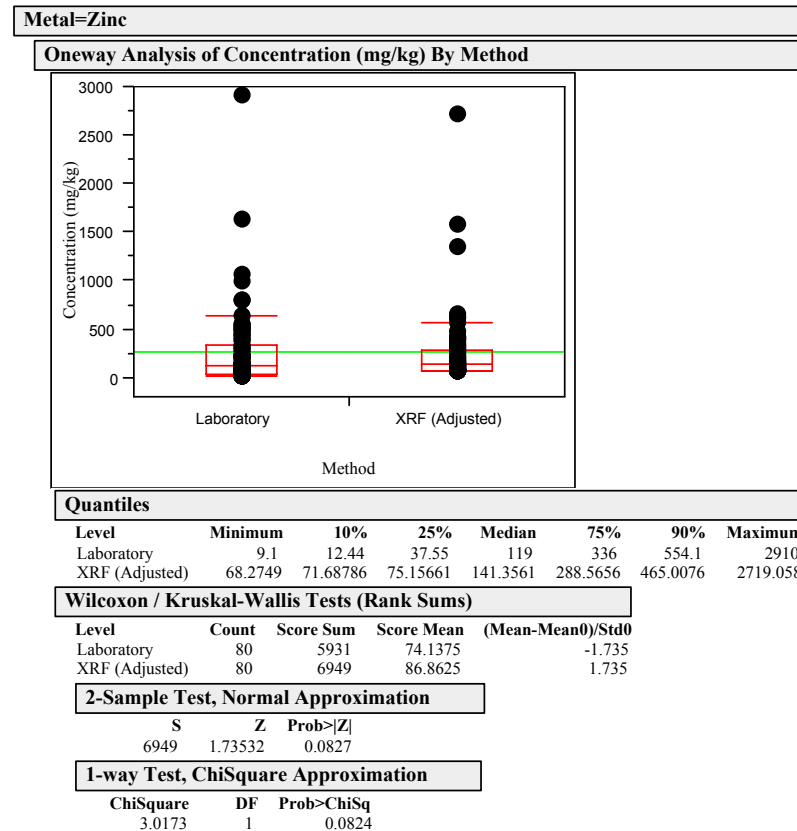
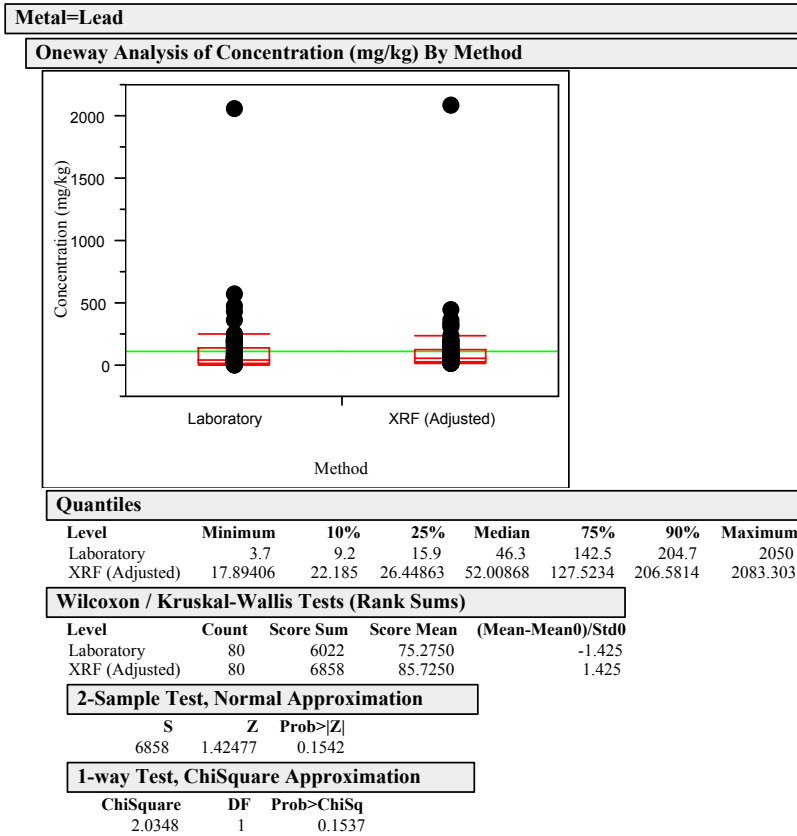
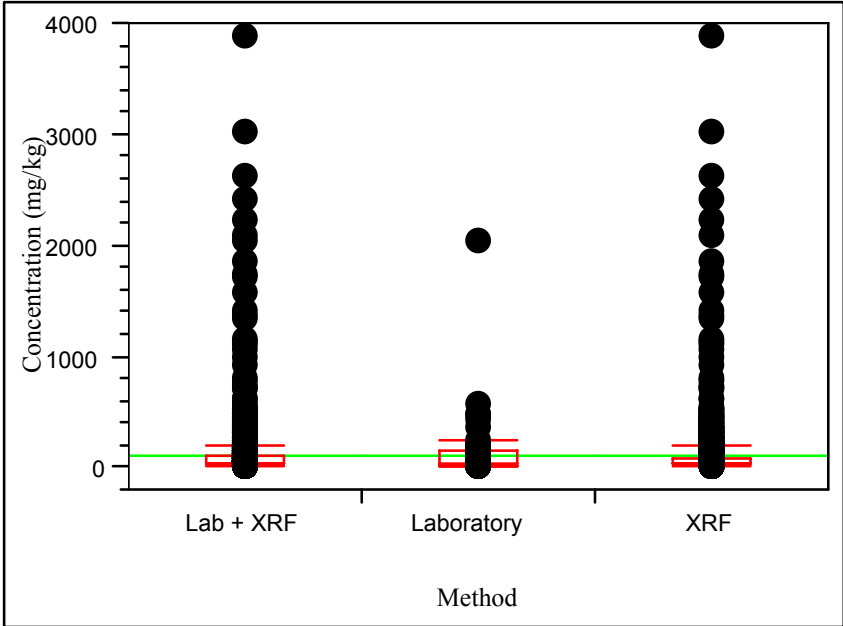


FIGURE 6

COMPARISON OF LEAD LABORATORY, ADJUSTED XRF, AND COMBINED LABORATORY AND ADJUSTED XRF CONCENTRATIONS

Metal=Lead (Adjusted Concentrations Used for XRF Data)
 Oneway Analysis of Concentration (mg/kg) By Method



Quantiles

Level	Minimum	10%	25%	Median	75%	90%	Maximum
Lab + XRF	3.7	22.51262	25.01528	37.61961	92.53668	216.4262	3886.274
Laboratory	3.7	9.04	14.85	42.6	147.5	205.4	2050
XRF	17.25702	22.82659	25.12904	37.02808	90.54466	219.0795	3886.274

Wilcoxon / Kruskal-Wallis Tests (Rank Sums)

Level	Count	Score Sum	Score Mean	(Mean-Mean0)/Std0
Lab + XRF	1013	1026675.5	1013.50	0.000
Laboratory	85	80903.5	951.81	-0.993
XRF	928	945772	1019.15	0.400

1-way Test, ChiSquare Approximation

ChiSquare	DF	Prob>ChiSq
1.0319	2	0.5969

TABLE 6

STATISTICAL COMPARISON OF LABORATORY AND XRF DATA USING LINEAR REGRESSION ANALYSIS
AND A TWO-POPULATION TEST

Chemical	Linear Regression Model ¹				Two-Population Test (Wilcoxon Rank Sum) ³		Conclusion of Two-Population Comparison Using the Wilcoxon Rank Sum Test
	Slope	Intercept	r ²	Prob>F ²	Z Score	Prob ⁴	
Arsenic	0.237	3.514	0.125	0.001	7.267	<0.001	* XRF (Median Concentration) > Laboratory (Median Concentration)
Chromium	0.086	42.930	0.466	<0.001	8.520	<0.001	* XRF (Median Concentration) > Laboratory (Median Concentration)
Cobalt	0.001	17.117	0.002	0.695	10.748	<0.001	* XRF (Median Concentration) > Laboratory (Median Concentration)
Copper	0.189	61.819	0.182	<0.001	0.886	0.190	* XRF (Median Concentration) ≤ Laboratory (Median Concentration)
Iron	0.432	5,577.766	0.787	<0.001	2.900	0.002	XRF (Median Concentration) > Laboratory (Median Concentration)
Lead	0.809	12.411	0.961	<0.001	0.469	0.319	* XRF (Median Concentration) ≤ Laboratory (Median Concentration)
Manganese	0.226	91.230	0.420	<0.001	5.837	<0.001	XRF (Median Concentration) > Laboratory (Median Concentration)
Mercury	0.004	0.049	0.029	0.128	11.019	<0.001	* XRF (Median Concentration) > Laboratory (Median Concentration)
Molybdenum	0.758	4.608	0.589	<0.001	0.808	0.210	* XRF (Median Concentration) ≤ Laboratory (Median Concentration)
Nickel	0.115	73.767	0.080	0.011	3.078	0.001	* XRF (Median Concentration) > Laboratory (Median Concentration)
Zinc	0.551	63.250	0.819	<0.001	0.271	0.393	XRF (Median Concentration) ≤ Laboratory (Median Concentration)

Notes:

- * Analysis was based on the detected and non-detected data combined. One-half of the reporting limit was substituted for all nondetect values.
- * Test result is suspect because of the low frequency of detection in either one or both populations being compared.
- r² Coefficient of determination, a measure of the strength of the linear relationship between the independent and dependent variables
- 1 XRF concentration is the independent variable; laboratory concentration is the dependent variable
- 2 Probability associated with test result. If Prob>F is less than or equal to 0.05 (five percent), then there is evidence of a significant regression effect.
- 3 Test of the one-sided null hypothesis that the median concentration of the XRF data is less than or equal to the median concentration of the laboratory data
- 4 Probability associated with test result. If Prob is less than or equal to 0.05 (five percent), then the null hypothesis is rejected.

generate laboratory-adjusted XRF data. The laboratory and adjusted XRF data were compared to each other using the nonparametric two-population WSR test for lead and zinc (Figure 6). If the regression-based adjustment were successful in providing laboratory-equivalent concentrations for the XRF data, then the relationships in the plots (Figure 6) would be 1:1. Since the two concentrations at each of the 80 locations should be identical (specifically within analytical limits for the precision of replicate measurements), this analysis provides a measure of the bias that would be introduced if adjusted XRF concentrations were used as true laboratory measurements. The lead plot indicates that the median concentrations are statistically equivalent, and that the two distributions are similar overall. However, in the case of zinc, the XRF data are higher. Following this analysis, Tetra Tech concluded that the field XRF data for lead could be used quantitatively for the RI and HHRA. For all other metals, the XRF data should be used for screening purposes only.

4.0 DATA EVALUATION PARAMETERS

Data were evaluated for acceptable quality and quantity based on the critical indicator parameters, including PARCCS. PARCCS parameters were reviewed for laboratory analytical data results and are discussed in the following sections.

4.1 PRECISION

Precision is the measure of the variability associated with an entire sampling and analysis process. It is the comparison among independent measurements as the result of repeated application of the same process under similar conditions. It is determined by analyzing field duplicate pairs, MSD pairs, and MD pairs. Precision is expressed as the RPD of a pair of values (or results). Acceptance criteria for each analytical methodology are presented in the QAPP (Tetra Tech 2003b). During the data validation process, field duplicate, MSD, and MD results were evaluated for compliance with acceptance criteria for precision for each analytical methodology. RPD evaluations are documented in individual DVRs for each SDG.

QAPP criteria specify that 10 percent of all sample matrices be submitted as field duplicates to the laboratory (Tetra Tech 2003b). Field duplicate pairs were collected, analyzed, and evaluated for each analysis performed on every sample matrix. Frequencies of field duplicate pairs submitted to the laboratory for analysis are provided in Table 7, and were shown to be 7.2 percent for the field project. The QAPP criterion for field duplicate precision is 50 percent RPD. Field duplicates exhibited poor precision for acetone, bromoform, bis(2-ethylhexyl)phthalate, benzo(g,h,i)perylene, carbazole,

TABLE 7
FIELD DUPLICATE FREQUENCY

Analysis	Soil			Ground Water			Surface Water			Sediment		
	Samples	FD	Percent	Samples	FD	Percent	Samples	FD	Percent	Samples	FD	Percent
CLP VOC	108	13	12.0	59	7	11.9	1	1	100	0	0	0
CLP SVOC	193	22	11.4	24	3	12.5	1	1	100	8	1	12.5
CLP Pest/PCB	124	14	11.3	0	0	0	0	0	0	8	1	12.5
CLP Metals	139	22	15.8	55	6	10.9	1	1	100	8	1	12.5
Metals & anions	102	16	15.7	59	6	10.2	0	0	0	0	0	0
Remedy suite	18	2	11.1	0	0	0	0	0	0	0	0	0
FBXRF	942	68	7.2 *	0	0	0	0	0	0	0	0	0

Notes:

* Frequency of 10 percent required by QAPP is not met for this parameter and matrix.

CLP Contract Laboratory Program
 FBXRF Field-based X-ray fluorescence
 FD Field duplicate
 PCB Polychlorinated biphenyl
 Pest Pesticides
 QAPP Quality assurance project plan
 SVOC Semivolatile organic compound
 VOC Volatile organic compound

phenanthrene, anthracene, pyrene, chrysene, benzo(a)anthracene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenzo(a,h)anthracene, fluoranthene, and benzo(b)fluoranthene, chromium, copper, iron, lead, manganese, nickel, zinc, endrin ketone, and 4,4'-DDT. Most of these exceedances were in soil samples, and generally indicate soil heterogeneity and the uneven distribution of contaminants in the matrix.

The QAPP states that the frequency criteria for MSD or MD pairs are 5 percent of the samples or one per analytical batch (Tetra Tech 2003b). MSD or MD samples were collected, analyzed, and evaluated for each analysis performed on every sample matrix. The frequencies in which MSDs or MDs were submitted to the laboratory for analysis are provided in Table 8. From Table 8, it is apparent that the MSD or MD frequency requirement was met for all analyses and matrices. QAPP criterion for MSD or MD precision is 50 percent (Tetra Tech 2003b) RPD; however, data were validated against CLP QC limits specified in applicable CLP protocols and the National Functional Guidelines (both of which are more stringent than 50 percent RPD). Compounds that exceeded CLP precision QC limits and resulted in qualified data include aluminum, arsenic, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, nickel, potassium, and zinc. Most of these exceedances were in soil samples and reflect a combination of both laboratory precision and the heterogeneity of the matrix.

4.2 ACCURACY

Accuracy is the degree to which a measurement agrees with its true value and is expressed as percent recovery; acceptance criteria for each analytical methodology are stated in the QAPP (Tetra Tech 2003b). Accuracy is assessed by comparing MS, LCS, and surrogate recoveries to associated QC limits. Through the process of data validation, MS, LCS, and surrogate recoveries were evaluated for compliance with acceptance criteria for accuracy for each applicable analytical methodology. Evaluations of percent recovery are documented in individual DVRs for each SDG.

The QAPP states that the frequency criteria for MSs are 5 percent of the samples or one per analytical batch (Tetra Tech 2003b). MS samples were collected, analyzed, and evaluated for each analysis performed on every sample matrix. Frequencies of MSs submitted to the laboratory for analysis are provided in Table 8. From Table 8, it is apparent that the MS frequency requirement was met for all analyses and matrices. QAPP criterion for MS accuracy is 50 to 150 percent for organic analyses and 75 to 125 percent for inorganic analyses (Tetra Tech 2003b); however, data was validated against CLP QC limits specified in applicable CLP protocols and the National Functional Guidelines (both of which are more stringent than QAPP criteria). Compounds that exceeded CLP QC limits and resulted in qualified

TABLE 8

MATRIX SPIKE, MATRIX SPIKE DUPLICATE, OR MATRIX DUPLICATE FREQUENCY

Analysis	Soil			Groundwater			Surface Water			Sediment		
	Samples	MS ¹	Percent	Samples	MS ¹	Percent	Samples	MS ¹	Percent	Samples	MS ¹	Percent
CLP VOC	108	9	8.3	59	5	8.5	1	1	100	0	0	0
CLP SVOC	193	12	6.2	24	2	8.3	1	1	100	8	1	12.5
CLP Pest/PCB	124	12	9.7	0	0	0	0	0	0	8	1	12.5
CLP Metals	139	21	15.1	55	5	9.0	1	1	100	8	1	12.5
Metals & anions	102	NR	NA	59	NR	NA	0	NR	NA	0	NR	NA
Remedy suite	18	2	11.1	0	0	0	0	0	0	0	0	0
FBXRF	942	NA ²	NA	0	0	0	0	0	0	0	0	0

Notes:

- 1 MSs and MSDs are required for organic tests, while MSs and MDs are required for metals and anions.
- 2 MSs and MDs are not applicable for FBXRF measurements because field duplicates suffice for precision determination.

CLP Contract Laboratory Program
 EPA U.S. Environmental Protection Agency
 FBXRF Field-based X-ray fluorescence
 MD Matrix duplicate
 MS Matrix spike
 MSD Matrix spike duplicate
 NA Not applicable
 NR Not reported by EPA Region 6 Laboratory
 PCB Polychlorinated biphenyl
 Pest Pesticides
 QAPP Quality assurance project plan
 SVOC Semivolatile organic compound
 VOC Volatile organic compound

data include heptachlor, γ -BHC, endrin, 4,4'-DDT, aluminum, antimony, arsenic, cobalt, copper, lead, manganese, selenium, silver, and vanadium. When recoveries were above or below the QC limits, a bias was assigned to the result (recoveries above the QC limits were qualified as biased high; while recoveries below the QC limits were qualified as biased low). In addition, the 4-nitrophenol result in one sediment sample was rejected due to zero percent recovery. The non-detected result may actually be a false negative; therefore, the result is unusable.

LCSs were analyzed for CLP low-concentration VOCs, low-concentration SVOCs, and metals only. LCS percent recoveries were within QC limits and analyzed at the required frequency.

Surrogate spikes were used in the analyses for VOCs, SVOCs, pesticides, and PCBs. Percent recoveries met QC criteria, with the following exceptions. Sample results for 2-hexanone and 4-methyl-2-pentanone in sample TB-17 were qualified as rejected (UR or R) because of very low surrogate recoveries. Results for 1,1,2,2-tetrachloroethane, 1,1-dichloroethane, 1,2-dibromo-3-chloropropane, 1,2-dibromoethane, bromochloromethane, bromoform, and dibromochloromethane in sample TWH6-01 were rejected (UR) due to very low surrogate recoveries. Quantitation limits for benzo(b)fluoranthene, benzo(a)pyrene, and dibenzo(a,h)anthracene were qualified as rejected (UR) due to extremely low surrogate recoveries (less than 10 percent recovery) in sample MW-13-01. Quantitation limits for the following compounds in the samples listed were qualified as rejected (UR) due to extremely low surrogate recoveries (less than 10 percent recovery).

- 3,3'-Dichlorobenzidine, 4-chloroaniline, and hexachlorocyclopentadiene in samples MW-05-01 and MW15-01
- 4-Chloroaniline, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, dibenzo(a,h)anthracene, hexachlorocyclopentadiene, and indeno(1,2,3-cd)pyrene in sample MW-13-01

Due to low surrogate recoveries, many SVOC compounds were qualified as estimated and possibly biased low, as well as, quantitation limits. Section 2.2 provides a complete listing of SVOC compounds for which data were qualified. In addition, positive results for endrin ketone, Aroclor 1254, and Aroclor 1260 were qualified as estimated and possibly biased high due to high surrogate recoveries.

Very low surrogate recoveries might indicate that the efficiency of purging VOCs from the samples to the instrument was not adequate or the efficiency of extracting SVOCs from the matrix was insufficient. The potential for false negatives exists; thus the reason for rejecting data discussed above.

4.3 REPRESENTATIVENESS

Representativeness is a qualitative parameter and is defined by the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or a process or environmental condition. Sample results were evaluated for representativeness by examining items related to sample collection, including chain-of-custody documentation, sample labeling, collection dates, and condition of the samples upon receipt at the laboratory. Laboratory procedures were also examined, including anomalies reported by the laboratory, either upon receipt of the samples at the laboratory or during analytical processes; adherence to recommended holding times of samples prior to analysis; calibration of laboratory instruments; adherence to analytical methods; and completeness of data package documentation. Any item that may have adversely affected the representativeness of the sample result is documented in the data validation narratives.

All samples were analyzed within the holding times specified by the methods except for aromatic VOCs in 30 samples. The holding time infraction was due to ineffective chemical preservation in VOC samples. The lack of preservation of VOC samples to a pH of less than 2 units caused the holding time for aromatic VOCs to be reduced from 14 days to 7 days. As such, these results were qualified as estimated and should be considered biased low. In addition, nitrate and nitrite results in four samples were qualified as estimated and possibly biased low because they were analyzed outside the technical holding time.

Initial and continuing calibrations generally met QC criteria for most VOC, SVOC, pesticide, PCB, metal, and inorganic analyses. However, VOC results for 1,2-dibromo-3-chloropropane, methyl acetate, and 2,4-dinitrophenol were rejected because of low calibration RRFs. Other sample results were qualified as estimated for VOCs, SVOCs, and metals analyses due to problems with instrument calibrations and calibration checks.

Equipment rinsate, trip blank, and laboratory method blank results were evaluated during the data validation process to determine whether equipment decontamination procedures (equipment rinsate), travel conditions (trip blank), or laboratory conditions (method blanks), may have affected sample results. Blank contamination was reported for 30 VOCs, 6 SVOCs, and 19 metals. The analytical results for the equipment rinsate, trip blanks, and laboratory method blanks are discussed in Section 2.0 for each analysis.

4.4 COMPLETENESS

Completeness is defined as the percentage of measurements judged to be valid. The validity of sample results is determined through the data validation process. All rejected (UR or R) sample results are considered to be incomplete. Data that are qualified as estimated (J) or estimated nondetected (UJ) are considered to be valid and usable. Completeness is calculated and reported for each method and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set.

The 90 percent completeness goal stated in the QAPP (Tetra Tech 2003b) was met; the summation of 47,462 total results includes 56 rejected points, resulting in a completeness of 99.9 percent for this project. A summary of rejected data is included in Table 9.

4.5 COMPARABILITY

Comparability of the data is a qualitative parameter that expresses the confidence with which one data set may be compared to another. Comparability of the data is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions, and using standardized reporting formats and data validation procedures. No method substitutions were observed that reduced the quality of the data for comparison purposes.

4.6 SENSITIVITY

Sensitivity is the measure of the signal from an instrument that represents an actual deflection or response above instrument noise. Analytical sensitivity is measured by the method detection limit (MDL) or IDL and reported with the necessary dilution factors, preparation factors, and dry-weight factors of an individual sample as the SQL. As stated in the QAPP (Tetra Tech 2003b), the required practical quantitation limits (PQL) for investigation sample analysis are equal to the CRQLs for SVOCs, VOCs, PCBs, and pesticides (although the latter is not a COPC) and the CRDLs for total metals analysis as provided in the EPA CLP protocols (EPA 1999a, 2000a, 2000b, and 2001).

Although (ideally) all of the detection limits outlined in Appendix A of the QAPP (Tetra Tech 2003b) would be below human health screening levels, available CLP detection limits are not always low enough to meet this goal. As discussed in the QAPP, in cases where the CLP detection limits were not low enough to meet screening levels, samples taken at the site were submitted to an EPA CLP lab, and a

TABLE 9
REJECTED RESULTS SUMMARY

Compound Name	Number of Rejected Results	Reason
Benzo(a)pyrene	2	Low surrogate recovery
Benzo(b)fluoranthene	2	Low surrogate recovery
Benzo(g,h,i)perylene	1	Low surrogate recovery
Benzo(k)fluoranthene	1	Low surrogate recovery
Bromochloromethane	1	Low surrogate recovery
Bromoform	1	Low surrogate recovery
4-Chloroaniline	3	Low surrogate recovery
Dibenzo(a,h)anthracene	2	Low surrogate recovery
Dibromochloromethane	1	Low surrogate recovery
1,2-Dibromo-3-chloropropane	5	Low calibration response
	1	Low surrogate recovery
1,2-Dibromoethane	1	Low surrogate recovery
3,3'-Dichlorobenzidine	2	Low surrogate recovery
2,4-Dinitrophenol	16	Low calibration response
	1	Low matrix spike recoveries
Hexachlorocyclopentadiene	3	Low surrogate recovery
2-Hexanone	1	Low surrogate recovery
Indeno(1,2,3-cd)pyrene	1	Low surrogate recovery
Mercury	1	Low calibration response
Methyl acetate	8	Low calibration response
4-Methyl-2-pentanone	1	Low surrogate recovery
1,1,2,2-Tetrachloroethane	1	Low surrogate recovery
Total Rejected Results	56	

flexibility clause was implemented in order to acquire the lowest possible detection limits. For Specific known exceptions to the desired detection limits were discussed during the DQO development, as noted in the QAPP. An assessment of the suitability of the detection limits achieved was determined for human health risk assessment (HHRA) and screening level ecological risk assessment (SLERA) data.

4.6.1 Human Health Risk Assessment

Because the first phase of the RI was intended to support an HHRA and determine the need for Phase II sampling, the collection of data that met the DQOs (i.e., were low enough to compare to human health benchmarks set forth in the tables in Appendix A of the QAPP [Tetra Tech 2003b]) was critical. Appropriately sensitive methods were selected for the analyses as indicated in the QAPP and as practical for analyses conducted by the EPA CLP. As stated in the QAPP, detection limits for the risk assessment were required to be below the EPA Region 6 Medium-Specific Screening Levels (MSSL) for residential exposure to soil. These risk-based screening levels are consistently based on a target risk of one in one million (for human cancer incidence) and (for human and ecological receptors) noncancer hazard quotients of 1. However, based on discussions with EPA, COPC detection limits that exceeded the carcinogenic screening values (10^{-6} cancer risk) would be acceptable if the detection limit falls within the 10^{-6} to 10^{-4} cancer risk range. Table 10 lists the analytes in soil samples that were found to have detection limits above risk-based screening levels for residential soil exposure. Table 11 lists the analytes in ground water samples that were found to have detection limits above risk-based screening levels for residential tap water exposure and/or MCLs. Note that carcinogens with SQLs below the 10^{-5} risk range were determined to be acceptable and not presented in Table 11.

Carcinogens that had SQLs that exceeded the 10^{-6} to 10^{-5} cancer risk range presented in Tables 10 and 11, but were still less than the upper bound of the total risk range for MDI (10^{-4}), included dibenzo(a,h)anthracene and n-nitroso-di-n-propylamine for soil exposure and 1,2-dibromo-3-chloropropane, 3,3'-dichlorobenzidine, atrazine, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, bis(2-ethylhexyl)phthalate, hexachlorobutadiene, indeno(1,2,3-cd)pyrene, and pentachlorophenol for ground water exposure.

In addition, analytes sampled in ground water that had SQLs exceeding the MSSLs for residential tap water exposure, but were less than the MCL for drinking water, included arsenic, trichloroethene, and vinyl chloride.

TABLE 10
SUMMARY OF COPCS WITH SQLs
ABOVE SOIL SCREENING LEVELS

COPC	Category	10 ⁻⁶ – 10 ⁻⁵ Cancer Risk Range ^a (mg/kg)	Noncancer ^a (mg/kg)	Highest SQL (mg/kg)	Number of Samples with SQLs Greater than the Required Detection Limit
2-Nitroaniline	N	N/A	3.7	4.6	1 of 165
Acetophenone	N	N/A	0.5	1.8	3 of 165
Dibenzo(a,h)anthracene	C	0.062 – 0.62	N/A	0.69	2 of 165
N-nitroso-di-n-propylamine	C	0.07 – 0.7	N/A	1.8	3 of 165

Notes:

Bolded COPCs indicate the highest SQL for this analyte exceeds the upper bound of the acceptable cancer risk range (10⁻⁴) or the noncancer threshold of 1.

aAs stated in the QAPP (Tetra Tech 2003b), detection limits below the EPA Region 6 Medium-Specific Screening Levels (MSSL; EPA 2002b) for residential exposure to soil were required for risk assessment purposes in order to meet the DQOs (see Section 1.0). These risk-based screening levels are based on a target risk of 10⁻⁶ (for carcinogens) and a noncancer hazard quotient of 1. For carcinogens, detection limits below the 10⁻⁵ cancer risk levels were acceptable based on discussions with EPA. Carcinogenic screening values based on a range of 10⁻⁶ to 10⁻⁵ for excess cancer risks are presented in this table.

C Carcinogen
COPC Contaminant of potential concern
mg/kg Milligram per kilogram
MSSL EPA Region 6 Medium-Specific Screening Level (EPA 2002b)
N Noncarcinogen
N/A Not applicable; no EPA Region 6 MSSL available
SQL Sample quantitation limit

TABLE 11

**SUMMARY OF COPCS WITH SQLs ABOVE
GROUND WATER SCREENING LEVELS**

COPC	MCL (ug/L)	Category	10 ⁻⁶ – 10 ⁻⁵ Cancer Risk Range ^a (ug/L)	Noncancer ^a (ug/L)	Highest SQL (ug/L)	No. of Samples with SQLs Greater than Required Detection Limit
1,2-Dibromo-3-chloropropane	0.2	C	0.048 – 0.48	N/A	0.5	5 of 27
1,2-Dibromoethane	N/A	C	0.00076 – 0.0076	N/A	0.5	27 of 27
2-Nitroaniline	N/A	N	N/A	2.2	36	27 of 27
3,3'-Dichlorobenzidine	N/A	C	0.15 – 1.5	N/A	9.1	24 of 25
Acetophenone	N/A	N	N/A	0.042	9.1	25 of 27
Antimony	6	N	N/A	15	60	5 of 27
Arsenic	10	C	0.0045 – 0.045	N/A	3*	1 of 27
Atrazine	3	C	0.3 – 3.0	N/A	9.1	26 of 27
Benzo(a)anthracene	N/A	C	0.092 – 0.92	N/A	5	5 of 27
Benzo(a)pyrene	0.2	C	0.0092 – 0.092	N/A	5	5 of 26
Benzo(b)fluoranthene	N/A	C	0.092 – 0.92	N/A	5	5 of 26
Bis(2-chloroethyl)ether	N/A	C	0.0098 – 0.098	N/A	9.1	27 of 27
Bis(2-ethylhexyl)phthalate	6	C	4.8 – 48	N/A	67	1 of 27
Dibenzo(a,h)anthracene	N/A	C	0.0092 – 0.092	N/A	5	5 of 26
Hexachlorobenzene	1	C	0.042 – 0.42	N/A	9.1	27 of 27
Hexachlorobutadiene	N/A	C	0.86 – 8.6	N/A	9.1	1 of 27
Indeno(1,2,3-cd)pyrene	N/A	C	0.092 – 0.92	N/A	9.1	26 of 26
Naphthalene	N/A	N	N/A	6.2	9.1	1 of 27
N-nitroso-di-n-propylamine	N/A	C	0.0096 – 0.096	N/A	9.1	27 of 27
Nitrobenzene	N/A	N	N/A	3.4	9.1	27 of 27
Pentachlorophenol	1	C	0.56 – 5.6	N/A	9.1	1 of 27
Trichloroethene	5	C	0.028 – 0.28	N/A	0.5*	4 of 27
Vinyl chloride	2	C	0.043 – 0.43	N/A	0.5*	5 of 27

Notes:

Bolded COPCs indicate the highest SQL for this analyte exceeds the upper bound of the acceptable cancer risk range (10⁻⁴) or the noncancer threshold of 1.

* The highest SQL for these analytes was above the residential MSSL for tap water; however, it is below the MCL .

TABLE 11 (Continued)

**SUMMARY OF COPCS WITH SQLs ABOVE
GROUND WATER SCREENING LEVELS**

Notes: (Continued)

a As stated in the QAPP (Tetra Tech 2003b), detection limits below the EPA Region 6 Medium-Specific Screening Levels (MSSL; EPA 2002b) for residential exposure to soil were required for risk assessment purposes in order to meet the DQOs (see Section 1.0). These risk-based screening levels are based on a target risk of 10^{-6} (for carcinogens) and a noncancer hazard quotient of 1. For carcinogens, detection limits below the 10^{-5} cancer risk levels were acceptable based on discussions with EPA. Carcinogenic screening values based on a range of 10^{-6} to 10^{-5} for excess cancer risks are presented in this table.

C Carcinogen
COPC Contaminant of potential concern
MCL Maximum Contaminant Level
MSSL EPA Region 6 Medium-Specific Screening Level (EPA 2002b)
N Noncarcinogen
N/A Not applicable; no EPA Region 6 MSSL or MCL available
SQL Sample quantitation limit
 μ/L Microgram per liter

One out of the 165 samples analyzed for 2-nitroaniline (0.6 percent) in soil had a detection limit greater than the 3.7 mg/kg MSSL. Because all of the other detection limits for this compound was below 3.7mg/kg and since none of these other samples had detected concentrations of 2-nitroaniline, it is not likely that the elevated detection limit for these few samples are masking concentrations above the MSSLs.

Also for soil, three out of the 165 samples analyzed for acetophenone (1.8 percent) had detection limits greater than the 0.5 mg/kg MSSL. However, because detected values were estimated as low as 0.011 mg/kg, it is likely that if the compound existed in the soil matrix it would have been qualified as an estimated concentration below the screening level. Therefore, it is unlikely that these elevated detection limits are of concern for this particular analyte.

In ground water, one of the 27 samples (3.6 percent) analyzed for naphthalene had detection limits greater than the 6.2 ug/L MSSL. Likewise, 5 of the 27 samples (18.5 percent) analyzed for dibenzo(a,h)anthracene had detection limits greater than the 0.092 ug/L upper bound of the cancer risk range. However, because detected values were estimated below the screening levels for each analyte, it is likely that if either of the compounds existed in the ground water matrix, it would have been qualified as an estimated concentration below the screening level. Therefore, it is unlikely that this elevated detection limit is of concern for either of these analytes.

Also in ground water, 5 out of the 27 samples analyzed for antimony (18.5 percent) had a detection limit greater than the 15 ug/L MSSL and the 6 ug/L MCL. However, because detected values were estimated as low as 1.2 ug/L, it is likely that if the compound existed in the soil matrix, it would have been qualified as an estimated concentration below the screening level. Therefore, it is unlikely that these elevated detection limits are of concern for antimony.

SQLs for the following chemicals were greater than the risk-based screening levels (i.e., greater than the upper bound of the acceptable cancer risk range [10⁻⁴] or a noncancer hazard threshold of 1) and MCLs, as appropriate, for samples in ground water:

- 1,2-Dibromoethane
- 2-Nitroaniline
- Acetophenone
- bis(2-Chloroethyl)ether
- Hexachlorobenzene

- n-Nitroso-di-n-propylamine
- Nitrobenzene

SQLs greater than the risk-based screening levels (or acceptable risk ranges) preclude estimating risk if present at concentrations below the SQL. Based on past investigations, none of these chemicals were identified as primary, site-related COPCs.

4.6.2 Ecological Risk Assessment

As with the HHRA, an attempt to achieve the detection limits set forth in Appendix A of the QAPP (see Tables A-1 through A-8) (Tetra Tech 2003b) was made, and appropriately sensitive methods were selected for the analyses as indicated in the QAPP and as practical for analyses conducted by the EPA CLP.

Specific known exceptions to the desired detection limits were discussed during the DQO development, as noted in the QAPP. An assessment of the suitability of the detection limits achieved was determined for SLERA data. Analytical testing of the soil and surface water samples collected during the RI yielded results with detection limits that were higher than the levels outlined in Appendix A of the QAPP. For soil, the following analytes had detection limits greater than the ecological benchmarks outlined in the QAPP:

- Molybdenum (5 of 55 samples analyzed [9.1 percent] had DLs greater than 2.0 mg/kg),
- Selenium (14 of 98 samples [14.3 percent] had DLs greater than 1.0 mg/kg),
- Thallium (40 of 98 samples [40.8 percent] had DLs greater than 1.0 mg/kg), and
- Vanadium (2 of 98 samples [2.0 percent] had DLs greater than 2.0 mg/kg).

However, because all of these analytes have detected values that were estimated below the required detection limits, it is likely that if any of these compounds existed in the soil matrix they would have been qualified as an estimated concentration below the screening level. Therefore, it is unlikely that these elevated detection limits are of concern for the SLERA.

In addition, for surface water, the following analytes had detection limits greater than the ecological benchmarks outlined in the QAPP:

- Anthracene (only two samples were analyzed and both had DLs greater than 0.3 ug/L),
- Benzo(a)pyrene (only two samples were analyzed and both had DLs greater than 0.014 ug/L),
- Lead (one of the two samples analyzed had a DL greater than 1.0 ug/L), and
- Silver (only two samples were analyzed and both had DLs greater than 0.8 ug/L).

A flexibility clause was implemented to obtain the lowest possible detection limits for surface water samples. Detection limits were achieved that were lower than the CRQLs, but still did not meet ecological screening levels for these analytes.

5.0 DATA QUALITY OBJECTIVES

Based on the data validation findings summarized in Section 2.0, data were either determined to be usable (unqualified or qualified as estimated) or rejected. All rejected data are usable for investigative purposes, specifically for answering the principal and secondary study questions derived from the project DQOs.

The following subsections discuss the usability of the data to conduct two major aspects of the investigation: (1) human health risk assessment and (2) ecological risk assessment.

5.1 HUMAN HEALTH AND ECOLOGICAL RISK ASSESSMENT USABILITY

As noted in the QAPP that accompanied the field sampling plan (FSP) (Tetra Tech 2003a), one of the goals of the RI field investigation and data collection efforts was to obtain “results of known quality that can support the ... human health and ecological risk assessments.” In applying the Data Usability for Risk Assessment guidance (EPA 1990, 1992a, 1992b), the following were key.

5.1.1 Chemicals of Potential Concern Targeted

As set forth in data usability guidance (EPA 1990, 1992a), industry-specific wastes, existing site data, and operational history were all considered in development of the MDI FSP (Tetra Tech 2003a) and QAPP (Tetra Tech 2003b). Because the FSP and QAPP set forth the DQOs for the MDI based on knowledge of past site operations and preliminary assessment findings, the chemicals of potential concern were

appropriately analyzed and targeted in the RI field investigation. Some specific findings with regard to this approach are discussed in the subsections below.

5.1.1.1 Polychlorinated Biphenyls

Some PCBs have dioxin-like properties that warrant their investigation using congener-specific analytical methods. However, like many special analytical techniques, this non-CLP analysis is expensive and not automatically warranted in a random grid sampling study design. For MDI, the site-specific FSP and QAPP (Tetra Tech 2003a; 2003b) assessed PCBs as total Aroclors (a CLP-available method) according to the agreed-upon DQOs for Phase I of the RI. Further, using biased sampling in suspected hot spots for PCB contamination, the media and locations to be sampled were conservatively targeted in an attempt to detect an Aroclor release above screening levels. No Aroclor samples were determined to be above screening levels for the SLERA or the HHRA. However, both risk assessments left the Aroclors as COPCs due to their bioaccumulative properties.

5.1.1.2 Tracer Metals (Molybdenum, Vanadium, and Nickel)

Because of the site-specific operational history of the site, molybdenum and nickel were thought to be candidate “tracer metals” indicative of process-related releases at MDI. Thus, special analyses for molybdenum were sought; nickel was present in a suite of standard inorganics under CLP analysis.

5.1.1.3 Hexavalent Chromium

To help in the toxicity effects assessment in the HHRA and the SLERA, not only was total chromium sampled, but hexavalent chromium (representing a potentially more toxic chromium valence state) was also sampled. Thus, special analyses for hexavalent chromium were performed; no hexavalent chromium was detected (to a detection limit of 1.0 mg/kg). This reduced the uncertainty associated with the interpretation of the “total chromium” values. This data confirmed that the “total chromium” assumptions inherent in HHRA toxicity values (a 1:6 ratio of hexavalent chromium to trivalent chromium) were conservative and appropriate.

5.1.1.4 Radiation

Based on the known operations at the MDI site and findings of slightly elevated radioactivity readings during a radiation survey (Enercon Services, Inc. 2003), sampling of a small, 25-square-foot area was conducted for additional screening. Based on the results of the soil screening results, Enercon Services, Inc. (Tetra Tech contractor) submitted six samples to Severn Trent Laboratories, Inc., in St. Louis, Missouri, for radium-226 and radium-228 analysis using EPA methods 903.0 MOD and 904 MOD, respectively (EPA 1984). The usability of radiological data is assessed for risk assessment in EPA guidance (EPA 1992b).

5.1.1.5 SVOCs

The “W” qualifier indicates that the SVOC result should be used with caution. The result was reported on a dry-weight basis although the sediment sample did not conform to the EPA Office of Water definition of a soil sample because of its high water content (greater than 70 percent moisture). High moisture sediment samples cannot be successfully analyzed by routine analytical methods, and additional analytical preparation steps are needed. Also, the aliquot extracted by the laboratory may not be representative because (1) of the low solid content and (2) detected results may be considered estimated. The estimated results may be biased high because of the adjustment for moisture content or biased low if the method was inadequate.

5.1.1.6 Pesticides

Although the pesticide data is usable and meets all quality control criteria, the “T” qualifier indicates that the identification is questionable because other commonly coexisting pesticides are absent. For example, the degradation products 4,4'-DDD and 4,4'-DDE are expected when 4,4-DDT is detected in a sample. Gamma-chlordane was detected in the sediment samples at a 0 to 6-inch maxima, but the coexisting pesticide alpha-chlordane was not detected. Therefore, the possibility of a false positive may exist as a chemically-similar compound may elute within the retention time windows for both columns. This may indicate that these data are not convincing evidence that the analyte in question was actually detected at the maximum concentration reported. In addition, pesticide contamination resulting from waste practices, waste disposal, or unauthorized or over-application of pesticides is not suspected at the MDI site. While these persistent pesticides may be present at low concentrations, no distinct release has been documented.

5.1.2 Library Search/TIC Reports

Although EPA (1990) discusses the utility of organic analysis tentatively identified compound (TIC) reports and mass spectral library search matching, this information was not available for the MDI site because the CLP program does not provide these services. As noted in EPA (1990), these types of data may be much less definitive than positive identifications. Therefore, although TICs were not addressed at the MDI site, the DQOs targeted the known or possible contaminants at the site, and therefore, no data gaps were concluded.

5.1.3 Media Variability

To minimize the effects of media variability issues on confidence in the analytical data, samples were collected from each medium of concern. For the HHRA, the media of concern were soil and ground water. For the SLERA, the media of concern were soil and surface water. Therefore, ground water samples were taken from temporary wells and monitoring wells, soil samples were taken at various locations around the site (including all areas of concern and identified hotspots), and a surface water sample was taken on site. In addition to these samples, field duplicates, MS/MSD samples, trip blanks, and equipment rinsates were taken in order to ensure that measurement error was reduced and to increase general confidence in the analytical results.

5.1.4 Sample Preparation

To determine whether the concentration of metals in the soil matrix had human health risk attributable to “respirable particles,” when sampling in the soil matrix, 45 of the 139 soil samples that were analyzed for total metals were passed through a Number 60 sieve and 94 were unsieved. The soil samples that were sieved were analyzed for the same constituents as their unsieved counterpart. For example, sample Q2-0.0-0.5U was unsieved and analyzed for total metals by CLP methods, while Q2-0.0-0.5 was the sieved sample and was also analyzed for total metals. The results of the sieved samples and their unsieved counterparts were then analyzed for statistical significant differences. Of the 23 metals tested, seven were higher in the unsieved soil (antimony, arsenic, calcium, lead, selenium, sodium, and thallium) and one was higher in the sieved soil (copper). The median concentrations were not statistically different for the other 15 metals.

Sampling for the SLERA included samples taken from on-site surface water. In order to evaluate the ecological impact of the dissolved metals, the samples that were analyzed were filtered in the field using a

0.45-micron filter. The filtering of the samples analyzed for metals intake allowed for metals to be evaluated as total metals and as dissolved metals.

5.1.5 Fixed Laboratory Versus Field Analyses

As discussed in Section 3.0, several soil samples were analyzed by using both XRF and laboratory resources in order to evaluate the effectiveness of the XRF data for the HHRA and SLERA. Statistical tests were conducted to find the correlation of the XRF and laboratory data for several metals. These tests revealed that only the XRF data for lead would be appropriate for quantitative use in the HHRA and the SLERA and that XRF data for all other metals should be used only for screening purposes.

5.1.6 Laboratory Performance Problems

Lab performance problems included issues with holding times and preservation, instrument calibration and calibration checks, method blanks, trip blanks, equipment rinsate blanks, and system monitoring compounds/surrogates. See Section 2.0 for a more detailed discussion of these problems.

6.0 CONCLUSIONS

Analytical results for this sampling event met overall project objectives for the quantity and quality of data required to support the decision-making process of this investigation. Only 56 data points out of 47,462 total measurements were rejected based on (1) poor responses in initial and continuing calibrations, (2) low surrogate recoveries, and (3) extremely low recoveries for MSs.

Data without qualifiers and data qualified as estimated are usable for purposes in supporting project objectives. Validated data were also found to be representative and comparable for all samples. Precision and accuracy were acceptable.

Statistical tests were conducted to find the correlation of the XRF and laboratory data for several metals. These tests revealed that only the XRF data for lead would be appropriate for quantitative use in the RI, HHRA, SLERA reports, and that XRF data for all other metals should be used only for screening purposes.

Carcinogens that had SQLs that exceeded the 10^{-6} to 10^{-5} cancer risk range, but were still less than the upper bound of the total risk range for MDI (10^{-4}), included dibenzo(a,h)anthracene and n-nitroso-di-n-

propylamine for soil exposure, and 1,2-dibromo-3-chloropropane, 3,3'-dichlorobenzidine, atrazine, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, bis(2-ethylhexyl)phthalate, hexachlorobutadiene, indeno(1,2,3-cd)pyrene, and pentachlorophenol for ground water exposure. In addition, analytes sampled in ground water that had SQLs that exceeded the MSSLs for residential tap water exposure, but were less than the MCL for drinking water, included arsenic, trichloroethene, and vinyl chloride. SQLs for 1,2-dibromoethane, 2-nitroaniline, acetophenone, bis(2-chloroethyl)ether, hexachlorobenzene, n-nitroso-di-n-propylamine, and nitrobenzene were greater than the risk-based screening levels (i.e., greater than the upper bound of the acceptable cancer risk range [10⁻⁴] or a noncancer hazard threshold of 1) and MCLs, as appropriate, for samples in ground water. SQLs greater than the risk-based screening levels (or acceptable risk ranges) preclude estimating risk if present at concentrations below the SQL. Based on past investigations, none of these chemicals were identified as primary, site-related COPCs.

Analytical testing of the soil and surface water samples collected during the RI yielded results with detection limits that were higher than the levels outlined in Appendix A of the QAPP. For soil, molybdenum, selenium, thallium, and vanadium had detection limits greater than the ecological benchmarks outlined in the QAPP. However, because all of these analytes have detected values that were estimated below the required detection limits, it is likely that if any of these compounds existed in the soil matrix they would have been qualified as an estimated concentration below the screening level. Therefore, it is unlikely that these elevated detection limits are of concern for the SLERA. A flexibility clause was implemented to obtain the lowest possible detection limits for surface water samples. Detection limits were achieved that were lower than the CRQLs, but still did not meet ecological screening levels for these analytes.

In order to achieve DQOs for this project, the QAPP (Tetra Tech 2003b) required that reporting limits for ground water and surface water media be lower than the standard CRQLs achievable by ICP/atomic emission spectroscopy. As such, the newest CLP protocol (ILM05.2) was requested for analysis of metals by ICP/mass spectrometry. In addition, a flexibility clause was implemented to set the project-required reporting limits as contractual limits. By exercising these variations on the basic protocol (ILM05.2), data for five analytes were not reported: calcium, iron, magnesium, potassium, and sodium. Because of the sensitivity of the ICP/mass spectrometry, mineral-related elements abundantly found in ground water and surface water samples are not as accurately measured and often require numerous serial dilutions that further enhance uncertainty of results. To this end, EPA does not require CLP laboratories to report these five analytes when conducting ICP/mass spectrometry analyses. These data are not available to support the risk assessment for ground water and surface water media.

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APPENDIX A

NON-CLP LABORATORY DATA VALIDATION REPORTS

ATTACHMENT A

CLP LABORATORY DATA VALIDATION REPORTS

ATTACHMENT B

EPA REGION 6 LABORATORY DATA REPORT NARRATIVES