

**DATA VALIDATION REPORT
FOR
FT. WINGATE B522 SAMPLING EVENT
Red Rock, New Mexico**

Samples Collected on July 12, 2010

Prepared by:

AMEC Earth & Environmental, Inc.
7376 SW Durham Road
Portland, Oregon 97224

1420102001.006

January 2011

Table of Contents

	<u>Page</u>
ACRONYMS AND ABBREVIATIONS.....	3
1.0 INTRODUCTION.....	5
2.0 EXECUTIVE SUMMARY.....	5
3.0 DATA VALIDATION METHODOLOGY.....	6
4.0 EXPLANATION OF DATA QUALITY INDICATORS.....	7
5.0 CHAIN OF CUSTODY AND SAMPLE RECEIPT CONDITION DOCUMENTATION.....	9
6.0 SPECIFIC DATA VALIDATION FINDINGS FOR EACH ANALYTICAL METHOD.....	9
6.1 Volatile Organic Compounds by EPA Method 8260B.....	9
6.1.1 Holding Times.....	9
6.1.2 Initial Calibration.....	9
6.1.3 Initial Calibration Verification.....	9
6.1.4 Continuing Calibration.....	10
6.1.5 Laboratory Blanks.....	10
6.1.6 Trip Blanks.....	11
6.1.7 Laboratory Control Sample Precision and Recovery.....	11
6.1.8 Matrix Spike/Matrix Spike Duplicate Precision and Recovery.....	11
6.1.9 Surrogate Recoveries.....	12
6.1.10 Internal Standard Recoveries.....	12
6.1.11 Analytical Procedures.....	12
6.2 Semivolatile Organic Compounds by EPA Method 8270.....	12
6.2.1 Holding Times.....	12
6.2.2 Initial Calibration.....	12
6.2.3 Initial Calibration Verification.....	12
6.2.4 Continuing Calibration.....	13
6.2.5 Laboratory Blanks.....	13
6.2.6 Surrogate Recoveries.....	13
6.2.7 Laboratory Control Sample Recovery.....	15
6.2.8 Matrix Spike/Matrix Spike Duplicate Precision and Recovery.....	15
6.2.9 Internal Standard Recoveries.....	17
6.2.10 Analytical Procedures.....	17
6.3 Nitrocellulose by TestAmerica SOP WS-WC-0050.....	17
6.3.1 Blanks.....	17
6.3.2 Laboratory Control Sample Recovery.....	17
6.3.3 Matrix Spike/Matrix Spike Duplicate Samples.....	17
6.3.4 Data Reporting and Analytical Procedure.....	18
6.4 Nitroaromatics and Nitramines by EPA SW-846 Method 8330.....	18
6.4.1 Holding Times.....	18
6.4.2 Initial Calibration.....	18
6.4.3 Initial Calibration Verification.....	18
6.4.4 Continuing Calibration.....	19

6.4.5	Blanks	19
6.4.6	Matrix Spike/Matrix Duplicate Recoveries and RPDs.....	19
6.4.7	Laboratory Control Sample Recovery.....	19
6.4.8	Surrogate Recoveries.....	19
6.4.9	Data Reporting	19
7.0	FIELD DUPLICATES.....	20
8.0	SUMMARY AND CONCLUSIONS.....	20
	REFERENCES.....	22

List of Tables

Table 1	Field Samples Submitted to TestAmerica and ALS
Table 2	Data Validation Qualifiers
Table 3	Field Duplicate Detected Results

ACRONYMS AND ABBREVIATIONS

µg/kg	micrograms per kilogram
%D	percent difference
%RSD	percent relative standard deviation
AMEC	AMEC Earth & Environmental, Inc.
CCV	continuing calibration verification
CLP	Contact Laboratory Program
COC	chain of custody
EPA	United States Environmental Protection Agency
ICAL	initial calibration
ICV	initial calibration verification
ID	sample identification
IS	internal standard
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
MDL	method detection limit
MEK	2-butanone
MIBK	4-methyl-2-pentanone
MS	matrix spike
MSD	matrix spike duplicate
QC	quality control
RF	response factor
RL	reporting limit
RPD	relative percent difference
SDG	sample delivery group
SOP	standard operating procedure
SVOCs	semivolatile organic compounds
TestAmerica	TestAmerica Laboratories, Inc.

VOCs

volatile organic compounds

1.0 INTRODUCTION

This data validation report covers 31 soil samples (including three field duplicates) and two aqueous trip blanks from the B522 area of the Fort Wingate site located in Red Rock, New Mexico. Samples were collected on July 13, 2010 and submitted to TestAmerica Laboratories, Inc. (TestAmerica) in North Canton, Ohio, where they were received on July 14, 2010. TestAmerica assigned the samples to sample delivery group (SDG) A0G140567 and analyzed them for volatile organic compounds (VOCs) by United States Environmental Protection Agency (EPA) SW846 Method 8260B and semivolatile organic compounds (SVOCs) by EPA SW846 Method 8270C. TestAmerica also shipped sample aliquots to the TestAmerica laboratory located in West Sacramento, California, where they were analyzed for nitrocellulose by TestAmerica standard operating procedure (SOP) WS-WC-0050 and nitroaromatics and nitramines by EPA SW846 Method 8330.

A list of these samples by field sample identification (ID) and TestAmerica sample ID is presented in Table 1.

2.0 EXECUTIVE SUMMARY

The data validation completed by AMEC Earth & Environmental, Inc. (AMEC) chemists indicate that the data from this event are generally usable and of acceptable quality with the following exceptions.

During validation, AMEC R qualified and rejected the nondetected 4-chloroaniline, 3,3'-dichlorobenzidine, 3-nitroaniline, and 4-nitroaniline results from sample 2160B522ED1SS-1-SO1 because of extremely low matrix spike (MS) recoveries. (Section 6.2.8)

During validation, AMEC U qualified the detected methylene chloride, bis(2-ethylhexyl)phthalate, and di-n-butyl phthalate results from sample 2160B522ED1SS-1-SO1 because these analytes were detected in the associated laboratory blanks at concentrations greater than 10% the concentrations detected in the sample. (Sections 6.1.5 and 6.2.5)

During validation, AMEC U qualified selected SVOC results because some, if not all, of the major ions were missing, resulting in poor chromatographic resolution. (Section 6.2.10)

The laboratory re-extracted sample 2160B522ED1SS-1-SO1 for the nitrocellulose analysis. The laboratory reported both the initial and the reanalysis data. AMEC evaluated both sets of data, and chose to report the higher concentration. The result considered non-reportable by AMEC was rejected. (Section 6.3.4)

Please note that a number of results, while considered usable, were qualified due to minor quality control (QC) anomalies. Specifically, AMEC qualified portions of the data because of low continuing calibration verification (CCV), laboratory control sample (LCS), MS, and surrogate recoveries; variability in the analytical results; and results reported between the method detection limit (MDL) and reporting limit (RL).

As stated above, these minor QC anomalies did not render the data unusable for use in site characterization or cleanup, but should be considered in the context of a data quality assessment if the data do not fall within expected ranges.

3.0 DATA VALIDATION METHODOLOGY

AMEC performed Level IV validation of 10% of the samples in this sample delivery group. This data validation was performed with reference to the requirements in the EPA Contract Laboratory Program (CLP) National Functional Guidelines for Superfund Organic Methods Data Review (6/08), the EPA CLP National Functional Guidelines for Superfund Inorganic Methods Data Review (1/10), the analytical methods referenced by the laboratory, and AMEC data validation procedures. Validation includes an assessment of the following:

- Chain of custody (COC) compliance
- Sample receipt
- Holding time compliance
- Reporting limits
- Calibrations
- Method blank results
- Surrogate Recoveries
- LCS/LCS duplicate (LCSD) precision and recoveries
- MS/MS duplicate (MSD) precision and recoveries
- Field QC results
- Internal Standard (IS) recoveries

Data that underwent validation are indicated on Table 1.

In general, it is important to recognize that no analytical data are guaranteed to be correct, even if all QC audits are passed. Strict QC serves to increase confidence in data, but any reported value may potentially contain error.

4.0 EXPLANATION OF DATA QUALITY INDICATORS

Summary explanations of the specific data quality indicators reviewed during this data validation are presented in the following table:

Data Quality Indicator	Description
LCS Recoveries	LCSs are aliquots of analyte-free water or clean solid matrix that are spiked with the analytes of interest for an analytical method, or a representative subset of those analytes. The spiked water or solid matrix is then processed through the same extraction, concentration, cleanup, and/or analytical procedures as the samples they accompany. LCS recovery is an indication of a laboratory's ability to successfully perform an analytical method in an interference-free matrix.
MS Recoveries	MSs and MSDs are prepared by adding known amounts of the analytes of interest for an analytical method, or a representative subset of those analytes, to an aliquot of sample. The spiked sample is then processed through the same extraction, concentration, cleanup, and analytical procedures as the unspiked samples in an analytical batch. MS recovery and precision are an indication of a laboratory's ability to successfully recover an analyte in the matrix of a specific sample or closely related sample matrices. It is important not to apply MS results for any specific sample to other samples without understanding how the sample matrices are related.
Surrogate Spike Recoveries	Surrogate spikes are used to evaluate accuracy, method performance, and extraction efficiency in each individual sample. Surrogate compounds are compounds not normally found in environmental samples, but which are similar to target analytes in chemical composition and behavior in the analytical process.

Data Quality Indicator	Description
<p>Blank Concentrations</p>	<p>Blank samples are aliquots of analyte free water or clean solid matrix that are used as negative controls to verify that the sample collection, storage, preparation, and analysis system does not produce false positive results.</p> <p>Laboratory blanks are processed by the laboratory using exactly the same procedures as the field samples. Target analytes should not be found in laboratory blanks.</p> <p>Trip blanks are sample vials of analyte free water that accompany the sample bottles to and from the collection site. The trip blank assesses potential ambient contamination from the site and laboratory. Target analytes should not be found in trip blanks.</p> <p>When target analytes are detected in blanks, analyte concentrations in associated samples greater than the RL but less than five times the concentration detected in the blank, or ten times the concentration detected in the blank for common laboratory contaminants, will be U qualified. Analyte concentrations between the MDL and RL, and less than five times (or again, ten times for common lab contaminants) the concentration detected in the blank will be U qualified at the RL concentration.</p>
<p>Laboratory Duplicates</p>	<p>Laboratory duplicate analysis verifies acceptable method precision by the laboratory at the time of preparation and analysis.</p>
<p>Internal Standards</p>	<p>IS are compounds that are added to a sample or extract after all preparatory steps are completed and before instrumental analysis. These compounds serve as standards for qualitative analysis using relative retention time and quantitative analysis using relative response factors (RFs). Methods that use IS calibration include requirements for changes in response to the IS relative to the initial calibration (ICAL).</p> <p>For EPA Methods 8260B and 8270C, IS response must fall between 50% and 200% of the response in the initial calibration.</p>
<p>Calibration</p>	<p>Instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. Calibration is verified at the beginning of the analytical run and on an ongoing basis.</p>

5.0 CHAIN OF CUSTODY AND SAMPLE RECEIPT CONDITION DOCUMENTATION

All samples were received at TestAmerica in good condition at temperatures less than the EPA-recommended maximum of 6 degrees Celsius.

All analyses were checked on the COCs for samples 2160B522DW01SS-1-SO4 and 2160B552EW7SS-1-SO4; however, TestAmerica only received two 40 milliliter vials of each sample. TestAmerica logged in the samples for VOC analysis only.

6.0 SPECIFIC DATA VALIDATION FINDINGS FOR EACH ANALYTICAL METHOD

Sections 6.1 through 6.4 contain narrative descriptions of the data validation findings and data quality limitations. Definitions of data qualifiers added during data validation and summaries of specific qualifiers added to each affected sample as a result of the data validation findings are presented in Table 2.

6.1 Volatile Organic Compounds by EPA Method 8260B

VOC results generated by TestAmerica may be considered usable with the limitations described in Sections 6.1.1 through 6.1.11.

6.1.1 Holding Times

All validated VOC samples were analyzed within The EPA-recommended maximum holding time of 14 days from collection.

6.1.2 Initial Calibration

ICALs met the method-specified criteria of $\leq 15\%$ relative standard deviation (%RSD) in response factor between levels, or coefficient of determination or correlation coefficients ≥ 0.990 .

6.1.3 Initial Calibration Verification

Initial calibration verification (ICV) standard recoveries were within the 70% to 130% guidance limits for unqualified data.

6.1.4 Continuing Calibration

CCV percent differences (%Ds) outside the method-specified $\pm 20\%$ limits are summarized below:

- %Ds for carbon disulfide and 1,2-dibromo-3-chloropropane were low at 78% and 79%, respectively, in the CCV associated with the analysis of samples 2160B522DW03SS-1-SO1, 2160B522DW03SS-1-SO2, and 2160B522DW04SS-1-SO1. AMEC UJ qualified the nondetected carbon disulfide and 1,2-dibromo-3-chloropropane results from these samples because of possibly low analytical bias.
- %Ds for acetone (69%), 2-butanone (MEK, 75%), chloromethane (75%), and 4-methyl-2-pentanone (MIBK, 79%) were low in the CCV associated with the analysis of sample 2160B522ED1SS-1-SO1. AMEC UJ qualified the nondetected results for these analytes in the associated sample because of possibly low analytical bias.

6.1.5 Laboratory Blanks

Target analytes were not detected in the laboratory blanks associated with the analysis of these samples, except as described below:

- Acetone was detected at a concentration of 3.4 micrograms per kilogram ($\mu\text{g}/\text{kg}$) in the laboratory blank associated with the analysis of samples 2160B522DW03SS-1-SO1, 2160B522DW03SS-1-SO2, and 2160B522DW04SS-1-SO1. Acetone was not detected in these samples, and data usability is not adversely affected.
- Trans-1,3-dichloropropene and methylene chloride were detected at concentrations of 0.86 $\mu\text{g}/\text{L}$ and 3.4 $\mu\text{g}/\text{L}$, respectively, in the laboratory blank associated with the analysis of sample 2160B522ED1SS-1-SO1. Specific limitations are summarized below:
 - Trans-1,3-dichloropropene was not detected in the associated sample, and data usability is not adversely affected.
 - AMEC U qualified the detected methylene chloride result from sample 2160B522ED1SS-1-SO1 at the RL of 5.5 $\mu\text{g}/\text{kg}$ because the concentration detected in the sample was less than the RL and less than ten times the concentration detected in the blank.

6.1.6 Trip Blanks

Target analytes were not detected in the trip blanks associated with these samples.

6.1.7 Laboratory Control Sample Precision and Recovery

Relative percent differences (RPDs) between LCS and LCSD results were less than the maximum-recommended 40% guidance limit for unqualified data. LCS recoveries outside the 80% to 120% guidance limits for unqualified data are described below:

- Bromoform (79%/77%) and carbon disulfide (78%-LCSD) recoveries were low in the LCS and/or LCSD associated with the analysis of samples 2160B522DW03SS-1-SO1, 2160B522DW03SS-1-SO2, and 2160B522DW04SS-1-SO1. AMEC UJ qualified the nondetected bromoform and carbon disulfide results from these samples because of possibly low analytical bias.
- Acetone (73%/56%), bromodichloromethane (73%-LCSD), bromoform (73%/59%), bromomethane (72%-LCSD), MEK (74%/58%), carbon disulfide (79%/70%), carbon tetrachloride (74%-LCSD), chloroethane (72%), chloroform (79%), chloromethane (69%/58%), 1,2-dibromo-3-chloropropane (63%-LCSD), 1,2-dichlorobenzene(78%-LCSD) ,1,3-dichlorobenzene (78%-LCSD), 1,4-dichlorobenzene (76%-LCSD), dichlorodifluoromethane (67%-LCSD), 1,1-dichloroethane (77%-LCSD), 1,2-dichloroethane (69%-LCSD), 1,2-dichloropropane (79%-LCSD), cis-1,3-dichloropropene (75%-LCSD), trans-1,3-dichloropropene (71%-LCSD), 2-hexanone (63%-LCSD), MIBK (64%-LCSD), 1,2,4-trichlorobenzene (76%-LCSD), 1,1,1-trichloroethane (77%-LCSD), trichloroethene (78%-LCSD), trichlorofluoromethane (73%-LCSD), and vinyl chloride (69%-LCSD) recoveries were low in the LCS and/or LCSD associated with the analysis of sample 2160B522ED1SS-1-SO1. AMEC UJ qualified the nondetected results for these analytes in this sample because of possibly low bias in the analytical results.

6.1.8 Matrix Spike/Matrix Spike Duplicate Precision and Recovery

TestAmerica performed MS on sample 2160B522ED1SS-1-SO1. RPDs between MS and MSD results were less than the maximum-recommended 40% guidance limit for unqualified data. MS recoveries outside the 70% to 130% guidance limits for unqualified data are described below:

Acetone (55%/59%), bromoform (60%/62%), bromomethane (65%-MSD), MEK (66%-MS), carbon disulfide (65%/66%), chloromethane (64%/62%),

1,2-dibromo-3-chloropropane (61%-MS), 2-hexanone (57%/56%), and 1,2,4-trichlorobenzene (57%/49%) recoveries were low in the MS and/or MSD performed on this sample. AMEC UJ qualified the nondetected results for these analytes in sample 2160B522ED1SS-1-SO1 because of possibly low analytical bias.

6.1.9 Surrogate Recoveries

Surrogate recoveries were within the 70% to 130% guidance limits for unqualified data.

6.1.10 Internal Standard Recoveries

IS recoveries were within the method-specified 50% to 200% limits for unqualified data.

6.1.11 Analytical Procedures

The laboratory J qualified detected results with concentrations between the RL and MDL. AMEC concurs that these results are quantitative estimates and J qualified these results in the final data table.

6.2 Semivolatile Organic Compounds by EPA Method 8270

SVOC results generated by TestAmerica may be considered usable with the limitations described in Sections 6.2.1 through 6.2.10.

6.2.1 Holding Times

All validated samples were extracted and analyzed within the EPA-recommended maximum holding time of 14 days from collection until extraction and 40 days from extraction until analysis.

6.2.2 Initial Calibration

ICALs met the method-specified criteria of $\leq 15\%$ RSD in response factor between levels, or coefficient of determination or correlation coefficients ≥ 0.990 .

6.2.3 Initial Calibration Verification

ICV standard recoveries were within the 70% to 130% guidance limits for unqualified data.

6.2.4 Continuing Calibration

CCV %Ds were within the method-specified $\pm 20\%$ limits for unqualified data.

6.2.5 Laboratory Blanks

SVOCs detected in the laboratory blanks associated with these samples are described below:

- Bis(2-ethylhexyl)phthalate was detected at a concentration of 100 $\mu\text{g}/\text{kg}$ in the laboratory blank associated with the analysis of samples 2160B522DW03SS-1-SO1, 2160B522DW03SS-1-SO2, and 2160B522DW04SS-1-SO1. AMEC U qualified the detected bis(2-ethylhexyl)phthalate results from samples 2160B522DW03SS-1-SO1 and 2160B522DW04SS-1-SO1 because the concentrations detected in the samples were less than ten times the concentration detected in the blank. (U-MB)
- Bis(2-ethylhexyl)phthalate and di-n-butyl phthalate were detected at concentrations of 30 $\mu\text{g}/\text{kg}$ and 31 $\mu\text{g}/\text{kg}$, respectively, in the laboratory blank associated with the analysis of sample 2160B522ED1SS-1-SO1. Specific limitations are summarized below:
 - AMEC U qualified the detected bis(2-ethylhexyl)phthalate result from sample 2160B522ED1SS-1-SO1 at the RL of 53 $\mu\text{g}/\text{kg}$ because the concentration detected in the sample was less than the RL and less than ten times the concentration detected in the blank.
 - AMEC U qualified the detected di-n-butyl phthalate result from sample 2160B522ED1SS-1-SO1 because the concentration detected in the sample was less than ten times the concentration detected in the blank.

6.2.6 Surrogate Recoveries

Surrogate recoveries outside the 70% to 130% guidance limits for unqualified data are described below:

- Samples 2160B522DW03SS-1-SO1 and 2160B522DW03SS-1-SO2 were analyzed at 1:5 dilutions, which made it difficult to fully evaluate matrix effects on surrogate recoveries.
- Recoveries of the surrogate compounds 2-fluorobiphenyl (48%), 2-fluorophenol (50%), phenol-d₅ (59%), 2,4,6-tribromophenol (59%), and nitrobenzene-d₅ (48%)

were low in sample 2160B522DW04SS-1-SO1. AMEC J qualified the detected results and UJ qualified the nondetected results from this sample because of possibly low analytical bias.

- Recoveries of the surrogate compounds 2-fluorobiphenyl (66%), 2-fluorophenol (54%), phenol-d₅ (56%), 2,4,6-tribromophenol (57%), and nitrobenzene-d₅ (52%) were low in sample 2160B522ED1SS-1-SO1. AMEC J qualified the detected results and UJ qualified the nondetected results from this sample because of possibly low analytical bias.
- Recoveries of the surrogate compounds 2-fluorobiphenyl (57%/57%), 2-fluorophenol (52%/54%), phenol-d₅ (53%/57%), 2,4,6-tribromophenol (55%/57%), nitrobenzene-d₅ (47%/50%), and terphenyl-d₁₄ (69%-MSD) were low in the MS and/or MSD performed on sample 2160B522ED1SS-1-SO1. AMEC does not qualify data from field samples based on MS surrogate recoveries, and data usability is not adversely affected.
- Recoveries of the surrogate compounds 2-fluorobiphenyl (66%), 2-fluorophenol (66%), phenol-d₅ (65%), 2,4,6-tribromophenol (37%), and nitrobenzene-d₅ (63%) were low in the laboratory blank associated with the analysis of samples 2160B522DW03SS-1-SO1, 2160B522DW03SS-1-SO2, and 2160B522DW04SS-1-SO1. AMEC does not qualify data from field samples based on surrogate recoveries in laboratory QC samples, and data usability is not adversely affected.
- Recoveries of the surrogate compounds 2-fluorobiphenyl (64%), 2-fluorophenol (65%), 2,4,6-tribromophenol (61%), and nitrobenzene-d₅ (58%) were low in the LCS associated with the analysis of samples 2160B522DW03SS-1-SO1, 2160B522DW03SS-1-SO2, and 2160B522DW04SS-1-SO1. AMEC does not qualify data from field samples based on surrogate recoveries in laboratory QC samples, and data usability is not adversely affected.
- Recoveries of the surrogate compounds 2-fluorobiphenyl (64%), phenol-d₅ (66%), 2,4,6-tribromophenol (37%), and nitrobenzene-d₅ (59%) were low in the laboratory blank associated with the analysis of sample 2160B522ED1SS-1-SO1. AMEC does not qualify data from field samples based on surrogate recoveries in laboratory QC samples, and data usability is not adversely affected.
- Recoveries of the surrogate compounds 2-fluorobiphenyl (64%), 2-fluorophenol (65%), 2,4,6-tribromophenol (61%), and nitrobenzene-d₅ (58%) were low in the laboratory blank associated with the analysis of sample 2160B522ED1SS-1-

SO1. AMEC does not qualify data from field samples based on surrogate recoveries in laboratory QC samples, and data usability is not adversely affected.

6.2.7 Laboratory Control Sample Recovery

LCS recoveries outside the 70% to 130% guidance limits for unqualified data are described below:

- 4-Chloroaniline (69%), 3,3'-dichlorobenzidine (61%), 4,6-dinitro-2-methylphenol (65%), 2,4-dinitrophenol (48%), hexachlorobutadiene (65%), and hexachloroethane (62%) recoveries were low in the LCS associated with the analysis of samples 2160B522DW03SS-1-SO1, 2160B522DW03SS-1-SO2, and 2160B522DW04SS-1-SO1. AMEC UJ qualified the nondetected results for these analytes in the associated samples because of possibly low analytical bias.
- Acenaphthene (59%), acenaphthylene (62%), bis(2-chloroethoxy)methane (58%), bis(2-chloroethyl)ether (57%), 4-chloroaniline (47%), 4-chloro-3-methylphenol (62%), 2-chloronaphthalene (60%), 2-chlorophenol (62%), dibenzofuran (65%), 3,3'-dichlorobenzidine (33%), 2,4-dichlorophenol (58%), 2,4-dimethylphenol (52%), 4,6-dinitro-2-methylphenol (15%), 2,4-dinitrophenol (15%), fluorene (69%), hexachlorobutadiene (53%), hexachloroethane (56%), isophorone (59%), 2-methylnaphthalene (59%), 2-methylphenol (62%), 4-methylphenol (59%), naphthalene (56%), nitrobenzene (59%), 2-nitrophenol (49%), 4-nitrophenol (46%), n-nitrosodi-n-propylamine (61%), pentachlorophenol (37%), phenol (60%), 2,4,5-trichlorophenol (56%), and 2,4,6-trichlorophenol (39%) recoveries were low in the LCS associated with the analysis of sample 2160B522ED1SS-1-SO1. AMEC UJ qualified the nondetected results for these analytes in the associated sample because of possibly low analytical bias.

6.2.8 Matrix Spike/Matrix Spike Duplicate Precision and Recovery

TestAmerica performed MS on sample 2160B522ED1SS-1-SO1. RPDs between MS and MSD results were less than the maximum-recommended 40% guidance limit for unqualified data. MS recoveries outside the 70% to 130% guidance limits for unqualified data are described below:

Acenaphthene (59%/60%), acenaphthylene (59%/60%), anthracene (63%/65%), benzo(ghi)perylene (69%-MS), benzo(a)pyrene (62%-MS), bis(2-chloroethoxy)methane (56%/60%), bis(2-chloroethyl)ether (45%/50%), 4-bromophenyl phenyl ether (69%/68%), carbazole (56%/60%), 4-chloroaniline

(0%/89%), 4-chloro-3-methylphenol (63%/65%), 2-chloronaphthalene (63%/64%), 2-chlorophenol (60%/58%), 4-chlorophenyl phenyl ether (67%/67%), chrysene (68%-MS), dibenz(a,h)anthracene (69%/68%), dibenzofuran (64%/65%), 3,3'-dichlorobenzidine (0%/0%), 2,4-dichlorophenol (63%/66%), diethyl phthalate (66%/65%), 2,4-dimethylphenol (59%/59%), dimethyl phthalate (65%/65%), 4,6-dinitro-2-methylphenol (40%/21%), 2,4-dinitrophenol (39%/27%), 2,4-dinitrotoluene (456%/0%), 2,6-dinitrotoluene (65%-MSD), fluorene (62%/63%), hexachlorobenzene (65%/64%), hexachlorobutadiene (55%/57%), hexachloroethane (40%/31%), indeno(1,2,3-cd)pyrene (67%-MS), isophorone (56%/57%), 2-methylnaphthalene (60%/64%), 2-methylphenol (56%/61%), 4-methylphenol (62%/63%), naphthalene (54%/57%), 2-nitroaniline (65%-MS), 3-nitroaniline (0%/31%), 4-nitroaniline (5.1%/30%), nitrobenzene (58%/55%), 2-nitrophenol (53%/51%), 4-nitrophenol (63%/61%), n-nitroso-di-n-propylamine (55%/61%), n-nitrosodiphenylamine (54%/21%), pentachlorophenol (51%/49%), phenanthrene (65%/64%), phenol (58%/61%), 2,4,5-trichlorophenol (62%/67%), and 2,4,6-trichlorophenol (64%/64%) were outside the guidance limits. Specific limitations are summarized below:

- AMEC R qualified and rejected the nondetected 4-chloroaniline, 3,3'-dichlorobenzidine, 3-nitroaniline, and 4-nitroaniline results from the spiked sample because of the extremely low (less than 10%) MS recoveries.
- The 2,4-dinitrotoluene concentration detected in the unspiked native sample, at 1,600 µg/kg, was greater than the spike concentration of 700 µg/kg, and the effect on data usability cannot be fully evaluated.
- AMEC J qualified the detected benzo(ghi)perylene, benzo(a)pyrene, chrysene, indeno(1,2,3-cd)pyrene, and n-nitrosodiphenylamine results from the spiked sample because of possibly low analytical bias.
- AMEC UJ qualified the nondetected acenaphthene, acenaphthylene, anthracene, bis(2-chloroethoxy)methane, bis(2-chloroethyl)ether, 4-bromophenyl phenyl ether, carbazole, 4-chloro-3-methylphenol, 2-chloronaphthalene, 2-chlorophenol, 4-chlorophenyl phenyl ether, dibenz(a,h)anthracene, dibenzofuran, 2,4-dichlorophenol, diethyl phthalate, 2,4-dimethylphenol, dimethyl phthalate, 4,6-dinitro-2-methylphenol, 2,4-dinitrophenol, 2,6-dinitrotoluene, fluorene, hexachlorobenzene, hexachlorobutadiene, hexachloroethane, isophorone, 2-methylnaphthalene, 2-methylphenol, 4-methylphenol, naphthalene, 2-nitroaniline, nitrobenzene, 2-nitrophenol, 4-nitrophenol, n-nitroso-di-n-propylamine, pentachlorophenol, phenanthrene, phenol, 2,4,5-trichlorophenol, and 2,4,6-trichlorophenol results from the unspiked sample because of possibly low analytical bias.

6.2.9 Internal Standard Recoveries

IS recoveries were within the method-specified 50% to 200% limits for unqualified data.

6.2.10 Analytical Procedures

The laboratory J qualified detected results with concentrations between the RL and MDL. AMEC concurs that these results are quantitative estimates and J qualified these results in the final data table.

AMEC U qualified the detected: benzo(b)fluoranthene and indeno(1,2,3-cd)pyrene results from sample 2160B522DW03SS-1-SO1, benzo(b)fluoranthene result from sample 2160B522DW03SS-1-SO2, benzo(a)anthracene result from sample 2160B522DW04SS-1-SO1, and benzo(k)fluoranthene result from sample 2160B522ED1SS-1-SO1 because some, if not all, of the major ions were missing, resulting in poor chromatographic resolution.

6.3 Nitrocellulose by TestAmerica SOP WS-WC-0050

Nitrocellulose results generated by TestAmerica may be considered usable with the limitations described in Sections 6.3.1 through Section 6.3.4.

6.3.1 Blanks

Nitrocellulose was not detected at concentrations greater than the RL in the laboratory blanks associated with the analysis of these samples.

6.3.2 Laboratory Control Sample Recovery

LCS recoveries were within the laboratory-established 34% to 115% limits for unqualified data.

6.3.3 Matrix Spike/Matrix Spike Duplicate Samples

TestAmerica performed MSs/MSDs on sample 2160B522ED1SS-1-SO1 twice. The initial background concentration in the unspiked native sample was reported as 10.4 milligrams per kilogram (mg/kg). The spike concentrations were 51.9 mg/kg in the MS and 53.1 mg/kg in the MSD. The measured concentration in the MS was 39.0 mg/kg, corresponding to 55% recovery. However, the measured concentration in the MSD was 278 mg/kg, corresponding to 504% recovery. Testamerica re-extracted and

reanalyzed sample 2160B522ED1SS-1-SO1 at a 1:10 dilution and the measured concentration was 205 mg/kg. When a MS and MSD were performed on the diluted sample, with spike concentrations of 53.1 mg/kg and 53.0 mg/kg, respectively, 39.4 mg/kg was detected in the MS and 68.5 mg/kg was detected in the MSD. The lack of reproducibility is likely due to a heterogeneous sample matrix, and AMEC chose not to qualify the data based on MS recoveries.

6.3.4 Data Reporting and Analytical Procedure

TestAmerica reported two nitrocellulose results for sample 2160B522ED1SS-1-SO1. The first reported concentration was 10.4 mg/kg, from an undiluted analysis, and the second reported concentration was 205 mg/kg, from a 1:10 dilution of the re-extracted sample. AMEC chose the higher of the two concentrations as the valid value, but J qualified the result as being estimated because of variability in the analytical results.

6.4 Nitroaromatics and Nitramines by EPA SW-846 Method 8330

Nitroaromatic and nitramine results generated by TestAmerica may be considered usable with the limitations described in Sections 6.4.1 through 6.4.9.

6.4.1 Holding Times

Samples were extracted for nitroaromatics and nitramines within the QAPP-specified maximum holding time of 14 days from collection for soils and analyzed within 40 days of extraction.

6.4.2 Initial Calibration

ICALs met the method-specified criteria of %RSDs $\leq 15\%$.

The confirmation column was not calibrated for 4-nitrotoluene. 4-Nitrotoluene was not detected in the field samples, and in AMEC's professional opinion, data usability is not adversely affected.

6.4.3 Initial Calibration Verification

ICV standard recoveries were within the method-specified 85% to 115% acceptance limits for unqualified data.

6.4.4 Continuing Calibration

All CCV standard recoveries were within the method-specified 85% to 115% acceptance limits for unqualified data.

6.4.5 Blanks

Nitroaromatics and nitramines were not detected in the laboratory blank associated with these samples.

6.4.6 Matrix Spike/Matrix Duplicate Recoveries and RPDs

TestAmerica performed MS on sample 2160B522ED1SS-1-SO1. RPDs between MS and MSD results were less than the maximum-recommended 40% guidance limit for unqualified data. MS recoveries outside the 70% to 130% guidance limits for unqualified data are described below:

2,4-Dinitrotoluene was detected at a concentration of 8.8 mg/kg in the unspiked native sample. The spike concentrations were 0.97 mg/kg in the MS and 1.0 mg/kg in the MSD. The measured concentration in the MS was 0.96 mg/kg and the measured concentration in the MSD was 1.2 mg/kg. The lack of reproducibility is likely due to a heterogeneous sample matrix. AMEC J qualified the result as being estimated because of variability in the analytical results.

6.4.7 Laboratory Control Sample Recovery

LCS recoveries were within the 70% to 130% guidance limits for unqualified data.

6.4.8 Surrogate Recoveries

Surrogate recoveries were within the 70% to 130% guidance limits for unqualified data

6.4.9 Data Reporting

The laboratory J qualified detected results with concentrations between the RL and MDL. AMEC concurs that these results are quantitative estimates and J qualified these results.

In sample 2160B522DW03SS-1-SO2 there was 40% RPD between the HMX results from the primary and confirmation columns. AMEC N qualified the detected HMX result from this sample as being presumptively identified because of the imprecision between the results from the two columns.

In sample 2160B522DW04SS-1-SO1 there was 61% RPD between the HMX results from the primary and confirmation columns. AMEC N qualified the detected HMX result from this sample as being presumptively identified because of the imprecision between the results from the two columns.

7.0 FIELD DUPLICATES

Samples collected as field duplicates are listed in Table 1. Field duplicate detected results are found in Table 3. The samples were labeled blindly, so the laboratory was not aware which samples were submitted in duplicate. Primary and duplicate results and the RPDs for the field duplicates are summarized in Table 3. With the exceptions listed in Table 3, precision values met the guidance limits for data usability of less than 40% RPD for soil for concentrations greater than five times their RL or \pm the RL for sample concentrations less than the RL.

8.0 SUMMARY AND CONCLUSIONS

AMEC's review indicates the data from this event are generally usable. The types of qualifications applied to the dataset include rejected, (R), estimated (J or UJ), presumptively identified (N), and nondetected (U) results.

During validation, AMEC R qualified and rejected the nondetected 4-chloroaniline, 3,3'-dichlorobenzidine, 3-nitroaniline, and 4-nitroaniline results from sample 2160B522ED1SS-1-SO1 because of extremely low matrix spike (MS) recoveries. (Section 6.2.8)

The remainder of the data is usable with the addition of the qualifiers listed in Table 2. Data that were qualified, but not rejected are summarized below.

AMEC J or UJ qualified data, as appropriate, when the associated CCV recoveries were low. (Section 6.1.4)

During validation, AMEC U qualified the detected methylene chloride, bis(2-ethylhexyl)phthalate, and di-n-butyl phthalate results from sample 2160B522ED1SS-1-SO1, and the bis(2-ethylhexyl)phthalate results from samples 21060B522DW03SS-1-SO1 and 2160B522DW04SS-1-SO1 because these analytes were detected in the associated laboratory blanks at concentrations greater than 10% the concentrations detected in the sample. (Sections 6.1.5 and 6.2.5)

During validation, AMEC J or UJ qualified data, as appropriate, when the associated LCS recoveries were low. (Sections 6.1.7 and 6.2.7)

During validation, AMEC J or UJ qualified results from sample 2160B522ED1SS-1-SO1 because of low MS and/or MSD recoveries. (Sections 6.1.8, 6.2.8, and 6.4.6)

During validation, AMEC J qualified results when the concentrations were between the MDL and RL. (Sections 6.1.11, 6.2.10, and 6.4.9)

During validation, AMEC J or UJ qualified, as appropriate, the SVOC results from samples 2160B522DW04SS-1-SO1 and 2160B522ED1SS-1-SO1 because of low surrogate recoveries. (Section 6.2.6)

During validation, AMEC U qualified selected SVOC results because some, if not all, of the major ions were missing, resulting in poor chromatographic resolution. (Section 6.2.10)

The laboratory re-extracted sample 2160B522ED1SS-1-SO1 for the nitrocellulose analysis. The laboratory reported both the initial and the reanalysis data. AMEC evaluated both sets of data, and chose to report the higher concentration. The result considered non-reportable by AMEC was rejected. (Section 6.3.4)

During validation, AMEC N qualified the detected HMX results from samples 2160B522DW03SS-1-SO1 and 2160B522DW04SS-1-SO1 because of imprecision between concentrations from the two analytical columns. (Section 6.4.9)

Data Completeness Assessment. AMEC reviewed 519 data points during the data validation. AMEC J qualified 30 results (5.8%), UJ qualified 155 results (30%), NJ qualified 2 results (0.39%), U qualified 5 results (0.96%), and R qualified and rejected 4 results (0.77%), meeting a 90% records as nondetected; and J or UJ qualified 486 (20%) records as estimated concentrations.

REFERENCES

EPA, 2010. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review, OSWER 9240.1-51, EPA 540-R-10-011.

EPA, 2008. *USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review*, EPA-540-R-08-01, June 2008.

EPA, 2007. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846 Final Update IV Revision 6, February 2007.

LIMITATIONS

This report was prepared exclusively for the PIKA International, Inc. by AMEC. The quality of information, conclusions, and estimates contained herein is consistent with the level of effort involved in AMEC services and based on: i) information available at the time of preparation, ii) data supplied by outside sources, and iii) the assumptions, conditions, and qualifications set forth in this report. This Data Validation/Review Report is intended to be used by the PIKA International, Inc. for the Fort Wingate site in Red Rock, New Mexico only, subject to the terms and conditions of its contract with AMEC. Any other use of, or reliance on, this report by any third party is at that party's sole risk.

TABLES