



**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12010479**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: February 8, 2012

Matrix: Groundwater, 1 sample

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L12010479

Sample Nos.: MP05-0112-1 (sic)

Comments: Sample number was recorded incorrectly on the COC and carried through the lab report; MP05-0112-1 should have been MPL05-0112-1. Field QC samples were not submitted for this SDG. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010*; *Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12010479  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	Ca 10x
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	Tl, Cd rr
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0	✓	✓	✓	✓	NA	✓	✓	NA	Cl 4x
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	NA
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	NA	✓	✓	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	NA	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on sample noted as internal laboratory QC (not a WSMR groundwater sample). Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance with one exception. Calcium percent recoveries (151 %R and 128 %R) were greater than the upper control limit. Data validation qualifiers were not assigned.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample was not submitted for this sample delivery group (SDG). MSD precision was found to be in compliance.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

## **IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant. Calcium was analyzed at a 10-times dilution in order to obtain a result within the calibration range.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in compliance with one exception. Cadmium was reanalyzed due to poor results on a low-level calibration check. The reanalysis was compliant.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance with one exception. Thallium was reanalyzed due to thallium detected in a continuing calibration blank. The reanalysis was compliant.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample identified as internal laboratory QC (not a WSMR groundwater sample). MS percent recovery for nickel was slightly less than criteria (79.5 percent versus 80 percent). All other analyte recoveries and duplicate relative percent differences were found to be in compliance. Data qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recovery and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Other**

A. LOQ were reviewed and found compliant

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were reported for this SDG.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A laboratory duplicate was analyzed as evidenced on the sample run log but the results were not reported in this SDG.

### **VIII. Other**

A. LOQ were reviewed and found compliant. Chloride concentration was determined from a 4-times dilution analysis.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show good agreement with 0.15 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A laboratory duplicate was analyzed on a non-project sample. Duplicate sample precision results were reviewed and found to be in compliance.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12010479, one sample MPL05-0112-1.**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG. Calcium (6010B) result is from a 10-times diluted analysis. Thallium and cadmium (6020) results are reported from a re-analysis due to QC failures during the original.

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG. Chloride (300.0) result is from a 4-times diluted analysis. All other anions are reported from the undiluted analysis.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.

COC No. A 28526

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Marietta, OH 45750

Microbac

Phone: 740-373-4071

Fax: 740-373-4835



CHAIN-OF-CUSTODY RECORD

Company Name: <i>Zia Shaw</i>						NUMBER OF CONTAINERS	Hold	Anions	Cond pH	PO <sub>4</sub>	Alkalinity	Total, Free, Amenable CN	Metals	NH <sub>3</sub> TOC NO <sub>3</sub> NO <sub>2</sub>	TSS TDS	TOTAL # (LAB USE)	Program	
Project Contact: <i>Mark Lyon</i>			Contact Phone #: <i>505-262-8920</i>														<input type="checkbox"/> CWA	<input type="checkbox"/> RCRA
Turn Around Requirements:			Location: <i>WSMR-STP</i>														<input type="checkbox"/> DOD	<input type="checkbox"/> AFCEE
Project ID: <i>STP</i>			Signature: <i>Bradley T. Davis</i>														<input type="checkbox"/> Other _____	
Sample I.D. No.	Comp	Grab	Date	Time	Matrix*													ADDITIONAL REQUIREMENTS
<i>MPLO6-0112-1</i>		<i>X</i>	<i>1-19-12</i>	<i>1146</i>	<i>W</i>	<i>7</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>				
<i>MPLO7-0112-1</i>		<i>X</i>	<i>1-19-12</i>	<i>1423</i>	<i>W</i>	<i>7</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>				
<i>MPLO7-0112-2</i>		<i>X</i>	<i>1-19-12</i>	<i>1423</i>	<i>W</i>	<i>7</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>	<i>X</i>				
Relinquished by: (Signature) <i>Bradley T. Davis</i>			Date	Time	Microbac OVD			221000021780			Date	Time	Received by: (Signature)					
Relinquished by: (Signature)			Date	Time	Received: 01/20/2012 11:44			By: BRENDA GREENWALT			Remarks:							
					<i>Brenda Greenwalt</i>													

\*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12010536**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: February 8, 2012

Matrix: Groundwater, 3 samples; 2 regular field samples, 1 field duplicate

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group: L12010536

Sample Nos.: MPL06-0112-1, MPL07-0112-1, MPL07-0112-2

Comments: MPL07-0112-2 is the field duplicate of MPL07-0112-1. The field duplicate sample is the only field QC submitted with these samples. Trip blank sample is not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

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- \* Temperature
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- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
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- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
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PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
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TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12010536  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	7
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0/SM4500-F-C	✓	✓	✓	✓	NA	✓	✓	NA	11
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	NA
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	NA	✓	✓	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	NA	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on sample noted as internal laboratory QC (not a WSMR groundwater sample). Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance with one exception. Calcium percent recoveries (130 %R and 158 %R) were greater than the upper control limit. Data validation qualifiers were not assigned.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). Relative percent differences (RPD) calculated when both sample and duplicate showed quantified results were acceptable. A summary of field duplicate precision is attached.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

## **IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant. Calcium was analyzed at 10-times dilution in all samples in order to obtain results within the calibration range.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in compliance with one exception. Cadmium recovery was slightly above criteria (126% versus 120%) in a low-level continuing calibration check. The analytical bias appeared high but as cadmium was not detected in any field sample, validation qualifiers were not assigned.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample identified as internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recoveries were acceptable for all target analytes.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). Relative percent differences (RPD) calculated when both sample and duplicate showed quantifiable results have precision for arsenic (39% RPD), selenium (37% RPD), and barium (5% RPD). Validation qualifiers are not assigned based solely on field duplicate variability. A summary of field duplicate precision is attached.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

**IX. Other**

A. LOQ were reviewed and found compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recovery and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample submitted with for this SDG showed the parent sample non-detect for mercury and the duplicate result as detected but less than the quantitation limit. Data validation qualifiers were not assigned.

### **VIII. Other**

A. LOQ were reviewed and found compliant.

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were reported for this SDG.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). Relative percent differences (RPD) calculated when both sample and duplicate showed quantified results were acceptable. A summary of field duplicate precision is attached.

### **VIII. Other**

A. LOQ were reviewed and found compliant. Chloride and sulfate in the field sample and field duplicate from MPL07 were analyzed at a 10X dilution in order to obtain results within the calibration curve. Fluoride was analyzed by an alternative method because of QC failures as narrated in the report.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show good agreement with 0.15 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). Relative percent differences (RPD) calculated when both sample and duplicate showed quantified results were acceptable. A summary of field duplicate precision is attached.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). Relative percent differences (RPD) calculated when both sample and duplicate showed quantified results were acceptable. A summary of field duplicate precision is attached.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). Relative percent differences (RPD) calculated when both sample and duplicate showed quantified results were acceptable. A summary of field duplicate precision is attached.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). Relative percent differences (RPD) calculated when both sample and duplicate showed quantified results were acceptable. A summary of field duplicate precision is attached.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

- A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

- A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

- A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

- A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

- A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

- A. A field duplicate sample was submitted with for this SDG. Analysis results in both samples were non-detect for ammonia.

### **VII. Other**

- A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). Relative percent differences (RPD) calculated when both sample and duplicate showed quantified results were acceptable. A summary of field duplicate precision is attached.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). The parameter was not detected in either sample. A summary of field duplicate precision is attached.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). Relative percent differences (RPD) calculated when both sample and duplicate showed quantified results were acceptable. A summary of field duplicate precision is attached.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). Relative percent differences (RPD) calculated when both sample and duplicate showed quantified results were acceptable. Results for total organic carbon were estimated concentrations detected less than the quantitation limits, in both the field sample and field duplicate. A summary of field duplicate precision is attached.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A field duplicate sample was submitted for this sample delivery group (SDG). The parameter was not detected in either sample. A summary of field duplicate precision is attached.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12010536**

**Three samples MPL06-0112-1, MPL07-0112-1, and MPL07-0112-2 (a field duplicate).**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG. Calcium (6010B) results are from a 10-times diluted analysis. .

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG. Chloride and sulfate (300.0) results are from 10-times diluted analyses in samples from MPL-07. Fluoride failed for QC measures by Method 300.0 and was analyzed by using the alternative method SM4500-F.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.

### III. Field Duplicate Precision

RPD calculated when both measurements exceed the limit of quantitation.

Method	Parameter	Field Sample MPL07-0112-1	Q	Field Duplicate MPL07-0112-2	Q	RPD
6010B	Beryllium	0.00200	U	0.00200	U	NC
6010B	Calcium	103		107		3.8%
6010B	Magnesium	17.6		17.7		0.6%
6010B	Manganese	0.0100	U	0.0100	U	NC
6010B	Potassium	3.23		3.27		1.2%
6010B	Sodium	43.4		44.8		3.2%
6010B	Tin	0.500	U	0.500	U	NC
6010B	Vanadium	0.0100	U	0.0100	U	NC
6010B	Zinc	0.0200	U	0.0200	U	NC
6020	Antimony	0.00100	U	0.00100	U	NC
6020	Arsenic	0.00584		0.00392		39.3%
6020	Barium	0.0521		0.0546		4.7%
6020	Cadmium	0.00600	U	0.00600	U	NC
6020	Chromium	0.00158		0.00155	J	NC
6020	Cobalt	0.00100	U	0.00100	U	NC
6020	Copper	0.00200	U	0.00200	U	NC
6020	Lead	0.00100	U	0.00100	U	NC
6020	Nickel	0.00296	J	0.00312	J	NC
6020	Selenium	0.02360		0.0163		36.6%
6020	Silver	0.00100	U	0.00100	U	NC
6020	Thallium	0.000200	U	0.000200	U	NC
7470A	Mercury	0.000200	U	0.000129	J	NC

300.0	Chloride	109		99.8		8.8%
300.0	Sulfate	129		120		7.2%
9040	pH	7.29		7.32		0.4%
310.2	Alkalinity, total	114		116		1.7%
9014	Cyanide	0.372		0.382		2.7%
120.1	Conductivity	878		888		1.1%
SM4500-F-C	Fluoride	0.127		0.123		3.2%
350.1	Ammonia, as N	0.100	U	0.100	U	NC
353.2	Nitrate-Nitrite, as N	11.9		12.4		4.1%
SM4500-P-E-20th	Orthophosphate	0.0500	U	0.0500	U	NC
160.1	Total Dissolved Solids	620		606		2.3%
415.1	Total Organic Carbon	0.928	J	0.849	J	NC

COC No. A 28524

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CHAIN-OF-CUSTODY RECORD

Company Name: <b>Zia / Shaw</b>						NUMBER OF CONTAINERS	Hold	Anions	Cond pH PO <sub>4</sub>	Alkalinity	Total, Free, Ammonia CN	Metals	NH <sub>3</sub> TOC NO <sub>3</sub> NO <sub>2</sub>	TSS TDS	TOTAL # (LAB USE)	Program	
Project Contact: <b>Mark Lyon</b>			Contact Phone #: <b>262-8920</b> <b>505-526-</b>													<input type="checkbox"/> CWA	<input type="checkbox"/> RCRA
Turn Around Requirements: <b>Normal</b>			Location: <b>WSMR</b>													<input type="checkbox"/> DOD	<input type="checkbox"/> AFCEE
Project ID: <b>STP</b>			Signature: <b>Bradley T. Davis</b>													<input type="checkbox"/> Other _____	ADDITIONAL REQUIREMENTS
Sampler (print): <b>Bradley T. Davis</b>																	
Sample I.D. No.	Comp	Grab	Date	Time	Matrix*												
MPL 18-0112-1		X	1-20-12	1030	W	7	X	X	X	X	X	X	X				
T40-0112-1		X	1-20-12	1210	W	7	X	X	X	X	X	X	X				
MPL 17-0112-1		X	1-20-12	1345	W	7	X	X	X	X	X	X	X				
Relinquished by (Signature): <b>Bradley T. Davis</b>						Date: <b>1-20-12</b>	Time: <b>1700</b>	Re (Si):	Microbac OVD Received: 01/21/2012 16:26 By: BOB BUCHANAN				221000021799	Date:	Time:	Received by (Signature):	
Relinquished by (Signature): <b>[Signature]</b>						Date:	Time:	Re (Si):	<b>[Signature]</b>				Remarks:				

\*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12010576**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: February 13, 2012

Matrix: Groundwater, 3 samples

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L12010576

Sample Nos.: MPL18-0112-1, T40-0112-1, and MPL17-0112-1

Comments: Field QC not included with this sample. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## **DATA VALIDATION QUALIFIER DEFINITIONS**

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12010576  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	Ca 10X
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0	✓	✓	✓	✓	NA	✓	✓	NA	11
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	NA
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	NA	✓	✓	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	NA	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on sample noted as internal laboratory QC (not a WSMR groundwater sample). Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance with one exception. Calcium percent recovery (126 %R) in the MSD was greater than the upper control limit. Data validation qualifiers were not assigned.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample was not submitted for this sample delivery group (SDG). MSD precision was found to be in compliance.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

## **IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant. Calcium was analyzed at a 10-times dilution in order to obtain a result within the calibration range.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample identified as internal laboratory QC (not a WSMR groundwater sample). Bias and precision measurements were within acceptable ranges.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance with one exception. Barium exceeded percent difference criteria however the serial dilution was not performed on a WSMR sample. Qualifiers were not assigned.

**IX. Other**

A. LOQ were reviewed and found compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recovery and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Other**

A. LOQ were reviewed and found compliant

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were not reported for this SDG.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A laboratory duplicate was analyzed as evidenced on the sample run log but the results were not reported in this SDG.

### **VIII. Other**

A. LOQ were reviewed and found compliant. Chloride concentration was determined from a 2-times dilution analysis in the sample from T40. Chloride, fluoride, and sulfate were all reported from a 4-times dilution of sample MPL17-0112-1. An undiluted analysis of MPL17-0112-1 should have been reported. The laboratory was requested to re-run fluoride and/or run fluoride by an alternative method.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the laboratory. The paired measurements show good agreement within 1.0 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A laboratory duplicate was analyzed on a non-project sample. Duplicate sample precision results were reviewed and found to be in compliance.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A matrix spike duplicate sample was noted on the laboratory run log. Those analyses were not on WSMR samples. Duplicate results were not available for review.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12010576  
Three samples MPL18-0112-1, T40-0112-1, and MPL17-0112-1.**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG. Calcium (6010B) results are from 10-times diluted analyses.

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG. Chloride and fluoride results in sample T40-0112-1 are from a 2-times diluted analysis. Chloride, fluoride, and sulfate analyses are from a 4-times diluted analysis of sample MPL17-0112-1. Fluoride was not detected at the elevated LOQ of 0.8 mg/L. The laboratory was requested to re-analyze for fluoride at a less dilution or by alternative method. Those results are pending.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes. Fluoride result in MPL17-0112-1 is pending re-analysis and may be confirmed or revised.



**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12010613**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: February 22, 2012

Matrix: Groundwater, 1 sample

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L12010613

Sample Nos.: MPL28-0112-1

Comments: Field QC not included with this sample. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12010613  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	✓
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	NA	✓	NA	NA	✓
Anions 300.0	✓	✓	✓	✓	NA	✓	NA	NA	✓
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	✓
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	✓
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	NA	✓	✓	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	NA	✓	NA	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	NA	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on sample noted as internal laboratory QC (not a WSMR groundwater sample). Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance except for calcium and sodium which recovered greater than the upper control limit. Data validation qualifiers were not assigned to WSMR samples.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample was not submitted for this sample delivery group (SDG). MSD precision was found to be in compliance.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

**IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 2°C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample identified as internal laboratory QC (not a WSMR groundwater sample). All analyte recoveries and duplicate relative percent differences were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recoveries were less than control limits and duplicate relative percent difference exceeded criteria. Validation qualifiers were not assigned to WSMR samples based only on MS/MSD results.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision exceeded acceptance limits.

### **VIII. Other**

A. LOQ were reviewed and found compliant. The post digest spike results were in compliance.

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were indicated on the run log analyzed on a non-WSMR sample. MS/MSD results were not reported.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A laboratory duplicate was analyzed as evidenced on the sample run log but the results were not reported in this SDG.

### **VIII. Other**

A. LOQ were reviewed and found compliant. The sample was analyzed at a 10-times dilution for chloride and sulfate. . High chloride concentrations would not allow for fluoride to be analyzed by ion chromatography. Fluoride was analyzed by alternative ion-specific electrode method SM4500-F-C. All QC measures were compliant in the fluoride analysis.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show agreement with 1 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A laboratory duplicate was analyzed on this WSMR sample. Duplicate sample precision results were reviewed and found to be in compliance.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. LCS duplicate and MSD were analyzed as indicated on the sample run log. Analyses were on non-WSMR samples and the results were not reported. Data validation qualifiers were not assigned.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12010613, one sample MPL28-0112-1.**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG. Chloride and sulfate (300.0) results are from a 10-times diluted analysis. Fluoride was analyzed by ion-selective electrode method.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

- A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

- A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.



**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12010651**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: February 22, 2012

Matrix: Groundwater, 1 sample

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L12010651

Sample Nos.: MPL16-0112-1

Comments: Additional sample volume was collected for MS/MSD requested. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010*; *Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12010651  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	6	✓	✓	✓	✓
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	9	✓	✓	NA	✓
Anions 300.0 / SM4500-F-C	✓	✓	✓	✓	✓	✓	✓	NA	✓
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	✓
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	✓
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	✓	✓	✓	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	✓	✓	✓	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	✓	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	✓	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	✓	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	✓	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	✓	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed on sample MPL16-0112-1. Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance with one exception. Calcium percent recoveries (134 %R and 170 %R) were greater than the upper control limit. Calcium result in MPL16-0112-1 is qualified with flag J+ indicating and estimated result with high bias.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample was not submitted for this sample delivery group (SDG). MSD precision was found to be in compliance.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

**IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant. Post digest spike results were within acceptance limits.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL16-0112-1. Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL16-0112-1. MS/MSD percent recoveries (76.5% and 46.5) and relative percent difference (48.8%) were out of compliance. Mercury was not detected in the original sample. Sample MPL16-0112-1 is qualified with “UJ” for estimated non-detect.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was out of compliance.

### **VIII. Other**

A. LOQ were reviewed and found compliant. The post-digest spike results were within acceptance limits.

## **ANIONS (Method 300.0 [9056] and SM4500-F-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant. Fluoride was analyzed using alternative selective-ion electrode method SM4500-F-C.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL16-0112-1. MS/MSD percent recoveries for chloride (86.8% and 87.4%) were noted less than the laboratory lower acceptance limit of 90% however the original sample concentration was greater than 4-times the spike level. Data validation qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. MSD and laboratory duplicate analyzed were within acceptance limits.

### **VIII. Other**

A. LOQ were reviewed and found compliant. Chloride and sulfate concentrations were determined from 4-times dilution analysis.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show good agreement with 0.30 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A laboratory duplicate was analyzed on a non-project sample. Duplicate sample precision results were reviewed and found to be in compliance.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL16-0112-1. MS/MSD percent recoveries and relative percent difference were compliant with acceptance limits.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VII. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VIII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL16-0112-1. MS/MSD percent recoveries and relative percent difference were compliant with acceptance limits except that the MSD percent recovery at 81.4% was less than the lower laboratory limit of 90%. Data validation qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VII. Duplicate**

A. The MSD results were reviewed and found to be compliant.

### **VIII. Other**

A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL16-0112-1. MS/MSD percent recoveries and relative percent difference were compliant with acceptance limits except that the MS percent recovery at 144% exceeded the upper lower laboratory limit of 110%. Data validation qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VII. Duplicate**

A. The laboratory control sample duplicate and MSD results were reviewed and found to be compliant.

### **VIII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL16-0112-1. MS/MSD percent recoveries and relative percent difference were compliant.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VII. Duplicate**

A. The laboratory control sample duplicate and MSD results were reviewed and found to be compliant.

### **VIII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. MS/MSD**

A. MS/MSD analyses were performed on sample MPL16-0112-1. MS/MSD percent recoveries and relative percent difference were compliant.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. The laboratory control sample duplicate and MSD results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL16-0112-1. MS/MSD percent recoveries and relative percent difference were compliant except the MS percent recovery at 83% was slightly less than the laboratory lower acceptance limit of 85%. Data validation qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VII. Duplicate**

A. The laboratory control sample duplicate and MSD results were reviewed and found to be compliant.

### **VIII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 3 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. MS/MSD**

A. MS/MSD analyses were performed on sample MPL16-0112-1. MS/MSD percent recoveries and relative percent difference were compliant.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. The laboratory control sample duplicate and MSD results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12010651, one sample MPL16-0112-1.**

### **Metals – Data Qualification Summary**

Calcium result in MPL16-0112-1 is qualified with flag J+ indicating and estimated result with high bias. Mercury was not detected in the original sample and is qualified with “UJ” for estimated non-detect. Qualifiers were assigned based on matrix spike results in sample MPL16-0112-1. All other metals analyses results were acceptable without qualification.

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG. Chloride and sulfate (300.0) result are from 4-times diluted analysis. Fluoride was analyzed by alternative ion-selective electrode method SM4500-F-C.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. Analysis results that were qualified during data validation are still usable for project purposes. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.



**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12010652**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: February 29, 2012

Matrix: Groundwater, 2 samples

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L12010652

Sample Nos.: MPL10-0112-1 and MPL13-0112-1

Comments: Field QC not included with this sample. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12010652  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	✓
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0/SM4500-F-C	✓	✓	✓	✓	NA	✓	✓	NA	Cl, SO4 2X
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	NA
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	NA	✓	NA	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	NA	✓	NA	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	NA	4X
Orthophosphate SM4500-P	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	NA	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on sample noted as internal laboratory QC (not a WSMR groundwater sample). Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance with one exception. Calcium percent recoveries (134 %R and 170 %R) were greater than the upper control limit. Data validation qualifiers were not assigned.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample was not submitted for this sample delivery group (SDG). MSD precision was found to be in compliance.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

**IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample identified as internal laboratory QC (not a WSMR groundwater sample). MS percent recoveries and duplicate relative percent differences were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recoveries were less than acceptance limits and duplicate relative percent difference exceeded specification. Data validation qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was out of compliance. Data validation qualifiers were not assigned as the MS/MSD sample was not identified as a WSMR sample.

### **VIII. Other**

A. LOQ were reviewed and found compliant. Post digest spike result was compliant.

## **ANIONS (Methods 300.0 [9056] and SM4500-F-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant. Fluoride was analyzed by ion-selective electrode method due to interferences from high concentrations of salts affecting Method 300.0.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were not reported for this Method 300.0 in this SDG. MS/MSD was compliant for fluoride analysis.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A laboratory duplicate was analyzed as evidenced on the sample run log but the results were not reported in this SDG.

### **VIII. Other**

A. LOQ were reviewed and found compliant. Chloride and sulfate concentrations were determined from 2-times dilution analyses.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show good agreement with 0.3 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A laboratory duplicate was analyzed on a non-project sample. Duplicate sample precision results were reviewed and found to be in compliance.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. A laboratory replicate was analyzed as indicated on the sample run log. The duplicate precision results were not reported.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A laboratory replicate was analyzed as indicated on the sample run log. The duplicate precision results were not reported.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant. Samples were analyzed at 4-times dilution.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12010652, two samples MPL10-0112-1 and MPL13-0112-1.**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG. Chloride and sulfate (300.0) results re are 2-times diluted analysis. Fluoride was analyzed by alternative ion-specific electrode method.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG. Nitrate + nitrite as N analyzed from 4-times diluted analyses.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.

COC No. A 28483

158 Starlite Drive  
Marietta, OH 45750

Microbac

Phone: 740-373-4071

Fax: 740-373-4835

CHAIN-OF-CUSTODY RECORD

Company Name: <b>Zia / SHAW</b>						NUMBER OF CONTAINERS	Hold	Anions	Cond. pH PO4	Alkalinity	Total Free Amenable CN	Metals	NH3 TOC NO3 NO2	TSS TDS	TOTAL # (LAB USE)	Program	
Project Contact: <b>Mark Lyon</b>			Contact Phone #: <b>505-262-8920</b>													<input type="checkbox"/> CWA	<input type="checkbox"/> RCRA
Turn Around Requirements: <b>Normal</b>			Location: <b>WSMR</b>													<input type="checkbox"/> DOD	<input type="checkbox"/> AFCEE
Project ID: <b>STP</b>			Signature: <b>Bradley T. Davis</b>													<input type="checkbox"/> Other _____	ADDITIONAL REQUIREMENTS
Sample I.D. No.	Comp	Grab	Date	Time	Matrix*												
<b>MPL 30-0112-1</b>		<b>X</b>	<b>1-25-12</b>	<b>1115</b>	<b>W</b>	<b>7</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>			
<b>MPL 29-0112-1</b>		<b>X</b>	<b>1-25-12</b>	<b>1350</b>	<b>W</b>	<b>7</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>			
<b>MPL-26-0112-1</b>		<b>X</b>	<b>1-25-12</b>	<b>1525</b>	<b>W</b>	<b>7</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>	<b>X</b>			
Relinquished by: (Signature) <b>Bradley T. Davis</b>						Date	Time	Received by: (Signature) _____									
Relinquished by: (Signature) _____						Date	Time	Remarks: _____									



Microbac OVD

221000021915

Received: 01/26/2012 10:42

By: BRENDA GREENWALT

*Brenda Greenwalt*

\*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12010686**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: February 29, 2012

Matrix: Groundwater, 3 sample

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L12010686

Sample Nos.: MPL26-0112-1, MPL29-0112-1, and MPL30-0112-1

Comments: Field QC not included with these sample. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## **DATA VALIDATION QUALIFIER DEFINITIONS**

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
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PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12010686  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	✓
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0	✓	✓	✓	✓	NA	✓	NA	NA	10
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	✓
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	NA	✓	NA	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	NA	4x-5x
Orthophosphate SM4500-P	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	NA	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on sample noted as internal laboratory QC (not a WSMR groundwater sample). Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance with one exception. Calcium percent recovery for the MSD, 155 % was greater than the upper control limit. The post digest spike was compliant for all metals. Data validation qualifiers were not assigned.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample was not submitted for this sample delivery group (SDG). MSD precision was found to be in compliance.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

**IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample identified as internal laboratory QC (not a WSMR groundwater sample). All analyte recoveries and duplicate relative percent differences were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recoveries and duplicate relative percent difference were out of compliance. As the sample was not identified as a WSMR groundwater sample data validation qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was out of compliance. Data validation qualifiers were not assigned.

### **VIII. Other**

A. LOQ were reviewed and found compliant.

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were not reported for this SDG.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A laboratory duplicate was analyzed as evidenced on the laboratory logs but the results were not reported in this SDG.

### **VIII. Other**

A. LOQ were reviewed and found compliant. Samples were analyzed at 10-times and 4-times dilutions in order to get chloride and sulfate concentrations within the calibration curves. Fluoride results that were non-detected at dilution and elevated LOQ were qualified "UN" for estimated non-detect.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show good agreement with 0.5 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A laboratory duplicate was analyzed on a non-project sample. Duplicate sample precision results were reviewed and found to be in compliance.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. Laboratory replicate analyses and matrix spike duplicate analyses were evidenced on the sample run log. None were WSMR groundwater samples. Laboratory analytical batch precision measures were not reported..

### **VII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 1°C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A laboratory replicate analyses was performed on MPL26-0112-1 as evidenced in the run log. Results for both analyses showed non-detect. Validation qualifiers were not assigned.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12010686, three samples MPL26-0112-1, MPL29-0112-1, and MPL30-0112-1.**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Anions – Data Qualification Summary**

Samples were analyzed at 10-times dilutions due to the chloride and sulfate concentrations exceeding the calibration range. Non-detect results for fluoride were qualified with “UN” flags indicating estimated LOQ. Fluoride was not re-analyzed at lower dilution nor analyzed using alternative ion-specific electrode.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

- A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

- A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.

COC No. A 28479

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CHAIN-OF-CUSTODY RECORD

Company Name: <b>Zia/Shaw</b>						NUMBER OF CONTAINERS	Hold	Anions	Cond. pH, PO <sub>4</sub>	Alkalinity	Total, Free, Ammonia CN	Metals	NO <sub>3</sub> NO <sub>2</sub>	TSS TDS	TOTAL # (LAB USE)	Program	
Project Contact: <b>Mark Lyon</b>		Contact Phone #: <b>505-262-8920</b>		<input type="checkbox"/> CWA	<input type="checkbox"/> RCRA												
Turn Around Requirements: <b>Normal</b>		Location: <b>WSMR</b>		<input type="checkbox"/> DOD	<input type="checkbox"/> AFCEE												
Project ID: <b>STP</b>		Signature: <b>Bradley T. Davis</b>		<input type="checkbox"/> Other _____													
Sampler (print): <b>Bradley T. Davis</b>		Signature: <b>Bradley T. Davis</b>		ADDITIONAL REQUIREMENTS													
Sample I.D. No.	Comp	Grab	Date	Time	Matrix*												
MPL 21-0112-1		X	1-27-12	1445	W	7		X	X	X	X	X	X	X			
MPL 21-0112-105		X	1-27-12	1445	W	7		X	X	X	X	X	X	X			
MPL 21-0112-105d		X	1-27-12	1445	W	7		X	X	X	X	X	X	X			
Relinquished by: (Signature) <b>Bradley T. Davis</b>						Date	Time	Received by: (Signature) <b>Jack Sheawa</b>						Date	Time	Received by: (Signature)	
Relinquished by: (Signature)						Date	Time	Received by: (Signature)						Remarks:			

\*Water (W), Soil (S), Solid Waste (SD), Unknown (X)



Microbac OVD

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Received: 01/28/2012 08:46

By: JACK SHEAWA

*Jack Sheawa*

**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12010767**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: March 8, 2012

Matrix: Groundwater, 1 sample

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group: L12010767

Sample Nos.: MPL21-0112-1

Comments: Extra, triplicate, sample volume was shipped to the laboratory for matrix spike and matrix spike duplicate analyses. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010*; *Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12010767  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	✓
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0	✓	✓	✓	✓	✓	✓	✓	NA	✓
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	NA
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	✓	✓	✓	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	✓	✓	✓	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	✓	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	✓	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	✓	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	✓	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	✓	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed on sample MPL21-0112-1. Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance with one exception. Calcium percent recoveries (76.7% and 68%) were less than the lower control limit. The parent sample concentration was greater than 4-times the spike level. Data validation qualifiers were not assigned.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample was not submitted for this sample delivery group (SDG). MSD precision was found to be in compliance.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

**IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL21-0112-1. MSD percent recovery and the resulting relative percent difference for copper exceeded laboratory acceptance limits. Copper in the parent sample was estimated less than the limit of quantitation, and the MS recovery for copper was compliant, as was copper recovery in the post digest spike. Data qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be compliant except for copper.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL21-0112-1. MS/MSD percent recovery and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Other**

A. LOQ were reviewed and found compliant

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL21-0112-1. Chloride and fluoride recovered slightly below the laboratory's lower acceptance limit of 90%. Data validation qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A laboratory duplicate of MPL21-0112-1 was analyzed. Duplicate precision was compliant.

### **VIII. Other**

A. LOQ were reviewed and found compliant.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show good agreement with 0.2 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A laboratory duplicate was analyzed on MPL21-0112-1 as evidenced on the run log. Duplicate sample precision results were reviewed and found to be in compliance.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL21-0112-1. MS/MSD bias and precision measures were within acceptance limits.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VII. Duplicate**

A. A MSD was analyzed and the results were found to be compliant.

### **VIII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL21-0112-1. Percent recoveries were less than the laboratory lower limit of 90% but greater than 80%. Data validation qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VII. Duplicate**

A. A MSD was analyzed. The results were reviewed and found to be compliant.

### **VIII. Other**

A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL21-0112-1. The MS percent recovery was less than the laboratory lower limit of 90% at 76.8%. MSD percent recovery and precision RPD were compliant. Data validation qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A LCS duplicate and MSD were analyzed. The results were reviewed and found to be compliant.

### **VIII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL21-0112-1. The percent recoveries and precision relative percent difference were reviewed and found to be compliant.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VII. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VIII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL21-0112-1. The percent recoveries and precision relative percent difference were reviewed and found to be compliant.

### **VI. Duplicate**

A. A LCS duplicate and MSD were analyzed and results found to be in compliance.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed on sample MPL21-0112-1. The percent recoveries and precision relative percent difference were reviewed and found to be compliant.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VII. Duplicate**

A. A LCS duplicate and MSD were analyzed. The results were reviewed and found to be compliant.

### **VIII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 4 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. MS/MSD**

A. MS/MSD analyses were performed on sample MPL21-0112-1. The percent recoveries and precision relative percent difference were reviewed and found to be compliant.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate and MSD were analyzed and results found to be in compliance.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12010767, one sample MPL21-0112-1 with extra volume submitted for MS/MSD analyses.**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG. All anions are reported from undiluted analysis.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.



**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12010798**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: March 8, 2012

Matrix: Groundwater, 1 sample

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group: L12010798

Sample Nos.: MPL22-0112-1

Comments: Field QC samples were not submitted for this SDG. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12010798  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	✓
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0	✓	✓	✓	✓	NA	✓	NA	NA	✓
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	NA
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	NA	✓	✓	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	NA	✓	NA	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	NA	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on sample noted as internal laboratory QC (not necessarily a WSMR groundwater sample). Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance with one exception. Calcium percent recoveries (76.7 %R and 68 %R) were less than the lower control limit. The parent sample concentration was greater than 4 times the matrix spike amount. Data validation qualifiers were not assigned.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. MSD precision was reviewed and found to be in compliance.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

**IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample identified as internal laboratory QC (not necessarily a WSMR groundwater sample). MS percent recoveries were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not necessarily a WSMR groundwater sample). MS/MSD percent recovery and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Other**

A. LOQ were reviewed and found compliant

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were not reported for this SDG.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A laboratory duplicate was analyzed as evidenced on the sample run log but the results were not reported in this SDG.

### **VIII. Other**

A. LOQ were reviewed and found compliant.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show good agreement with 0.25 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A laboratory duplicate was analyzed on a non-project sample. Duplicate sample precision results were reviewed and found to be in compliance.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. A sample duplicate was analyzed as evidenced on the laboratory run log and data sheet. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. Laboratory duplicate sample analyses were evidenced on the run log data sheet but not reported in this SDG..

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12010798, one sample MPL22-0112-1.**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG. Chloride (300.0) result is from a 4-times diluted analysis. **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.



COC No. A 28492

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CHAIN-OF-CUSTODY RECORD

Company Name: <b>Zia / SHAW</b>						NUMBER OF CONTAINERS	Hold	Anions	Cond pH PO4	Alkalinity	Total Free Ammonia CN	Metals	NH <sub>3</sub> TOC NO <sub>2</sub> NO <sub>3</sub>	TSS TDS	VOCs	RADs	PCBs	PAHs	Total Phenols	Al, B, Fe, Mn, Mo	TOTAL # (LAB USE)	Program					
Project Contact: <b>Mark Lyon</b>			Contact Phone #: <b>505-262-8920</b>																			<input type="checkbox"/> CWA					
Turn Around Requirements: <b>Normal</b>			Location: <b>WSMR</b>																			<input type="checkbox"/> RCRA					
Project ID: <b>STP</b>			Signature: <b>Bradley Davis</b>																			<input type="checkbox"/> DOD					
Sampler (print): <b>Bradley Davis</b>			Signature: <b>Bradley Davis</b>			<input type="checkbox"/> AFCEE																					
Sample I.D. No.	Comp	Grab	Date	Time	Matrix*																ADDITIONAL REQUIREMENTS						
MPL4-0112-1		X	1-30-12	1355	W	16	X	X	X	X	X	X	X	X	X	X	X	X	X								
MPL4-0112-MS		X	1-30-12	1355	W	16	X	X	X	X	X	X	X	X	X	X	X	X	X								
MPL4-0112-MSD		X	1-30-12	1355	W	16	X	X	X	X	X	X	X	X	X	X	X	X	X								
MPL2-0112-1		X	1-30-12	1705	W	16	X	X	X	X	X	X	X	X	X	X	X	X	X								
MPL3-0112-1		X	1-31-12	1015	W	16	X	X	X	X	X	X	X	X	X	X	X	X	X								
MPL3-0112-2		X	1-31-12	1015	W	16	X	X	X	X	X	X	X	X	X	X	X	X	X								
MPL1-0112-1		X	1-31-12	1500	W	16	X	X	X	X	X	X	X	X	X	X	X	X	X								

Microbac OVD  
 Received: 02/01/2012 10:07  
 By: CARA STRICKLER

221000022012

Relinquished by: **Bradley Davis** Date: **1-31-12** Time: **1700**  
 Relinquished by: (Signature) Date Time

Date Time Received by: (Signature)

**Cara Strickler**

Remarks:

\*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12020016**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: February 15, 2012

Matrix: Groundwater; 4 groundwater samples, 1 field duplicate, and extra volume for MS/MSD

Parameters: Volatile Organic Compounds 8260B  
Semivolatile Organic Compounds, Polycyclic Aromatic Hydrocarbons, 8270C  
Polychlorinated Biphenyls 8082  
Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Phenols 420.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group: L12020016

Sample Nos.: MPL4-0112-1 (MS/MSD requested), MPL2-0112-1, MPL3-0112-1, MPL3-0112-2 (Field Duplicate), MPL1-0112-1

Comments: MS/MSD requested on field sample MPL4-0112-1. Field duplicate submitted. A trip blank sample was submitted to the laboratory along with the field samples but the trip blank was not recorded on the COC.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010*; *Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12020016  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Volatile Organics 8260B	✓	✓	✓	✓	✓	✓	✓	NA	✓
PAH 8270C	✓	✓	✓	✓	✓	✓	✓	NA	✓
PCB 8082	✓	✓	✓	✓	✓	✓	✓	NA	NA
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	✓
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0	✓	✓	✓	✓	18	✓	✓	NA	18
pH 9040C	✓	✓	✓	NA	✓	✓	✓	NA	NA
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	✓	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	✓	✓	✓	NA	✓
Conductivity 120.1	✓	✓	NA	✓	✓	✓	✓	NA	✓
Phenols 420.1	✓	✓	✓	✓	✓	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	✓	✓	✓	NA	NA
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	✓	✓	✓	NA	NA
Orthophosphate SM4500-P	✓	✓	✓	✓	✓	✓	✓	NA	NA
Total Dissolved Solids 160.1	✓	✓	NA	✓	✓	✓	✓	NA	NA

<b>Total Organic Carbon 415.1</b>	✓	✓	✓	✓	✓	✓	✓	NA	NA
<b>Total Suspended Solids 160.2</b>	✓	✓	NA	✓	✓	✓	✓	NA	NA

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

## DATA ASSESSMENT

### Volatile Organic Compounds (Method 8260B)

#### I. Temperature

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

#### II. Holding Times

A. The analysis holding times were reviewed and found to be in compliance.

#### III. Calibration

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance with one exception for bromomethane in one CCV. Validation qualifiers were not assigned as the CCV bias was slightly more than the upper criteria and the compound was not detected in any samples, however the laboratory re-analyzed MPL4-0112-1 and associated MS/MSD to achieve compliance. The laboratory reported both sets of results. Data review selected the re-analyses as the reportable results.

#### IV. Blanks

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

#### V. Surrogate Recoveries

A. Surrogate spike recoveries were reviewed and found to be in compliance.

#### VI. Matrix Spike / Matrix Spike Duplicate (MS/MSD)

A. MS/MSD analyses were performed on WSMR groundwater sample MPL4-0112-1. Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance.

#### VII. Laboratory Control Sample (LCS)

A. The LCS results were reviewed and found to be in compliance.

#### VIII. Duplicate

A. A field duplicate sample, MPL3-0112-2 was submitted for this sample delivery group (SDG). Field duplicate precision was acceptable and there were no compounds detected at concentrations greater than the applicable LOQ.

**IX. Internal Standards Performance**

A. Internal standards retention times were reviewed and found to be in compliance.

## **DATA ASSESSMENT**

### **Semi-volatile Organic Compounds Polycyclic Aromatic Hydrocarbons (PAH) (Method 8270C)**

#### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

#### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

#### **III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

#### **IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

#### **V. Surrogate Recoveries**

A. Surrogate spike recoveries were reviewed and found to be in compliance.

#### **VI. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed on WSMR groundwater sample MPL4-0112-1. Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance with minor exceptions. Four compounds recovered slightly above the upper acceptance criteria. Validation qualifiers were not assigned because the bias shown was high and the compounds were not detected in any field sample.

#### **VII. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance with minor exceptions. Chrysene and Benzo-a-pyrene recoveries were slightly more than the upper acceptance limit. Validation qualifiers were not assigned because the bias shown was high and the compounds were not detected in any field sample.

### **VIII. Duplicate**

A. A field duplicate sample, MPL3-0112-2 was submitted for this sample delivery group (SDG). Field duplicate precision was acceptable and there were no compounds detected at concentrations greater than the applicable LOQ.

### **IX. Internal Standards Performance**

A. Internal standards retention times were reviewed and found to be in compliance.

## **DATA ASSESSMENT**

### **Polychlorinated Biphenyls (PCB) (Method 8082A)**

#### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

#### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

#### **III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

#### **IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

#### **V. Surrogate Recoveries**

A. Surrogate spike recoveries were reviewed and found to be in compliance.

#### **VI. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed on WSMR groundwater sample MPL4-0112-1. Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance.

#### **VII. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

#### **VIII. Duplicate**

A. A field duplicate sample, MPL3-0112-2 was submitted for this sample delivery group (SDG). Field duplicate precision was acceptable and there were no compounds detected at concentrations greater than the applicable LOQ.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on WSMR groundwater sample MPL4-0112-1. Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance except for calcium. Calcium spike recoveries were 70.2 percent and 129 percent, however the parent sample concentration exceeded 5-times the spike level. Data validation qualifiers were not assigned.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was generally acceptable and RPD was calculated for elements when both sample and duplicate sample results were greater than the LOQ. Percent RPD for aluminum (124%), iron (66%), and zinc (87%) appear excessive however in each case one or both results were less than 5-times the LOQ. Data validation qualifiers were not assigned based on field duplicate results.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

**IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in compliance with exceptions for cadmium and thallium. These metals were reanalyzed due to poor results on a low-level calibration check. The reanalysis were compliant.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance with one exception. Thallium was reanalyzed due to thallium detected in a continuing calibration blank. The reanalysis was compliant.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on WSMR groundwater sample MPL4-0112-1. Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was generally acceptable and RPD was calculated for elements when both sample and duplicate sample results were greater than the LOQ. Percent RPD for manganese (117%) appears excessive however one or both results were less than 5-times the LOQ. Data validation qualifiers were not assigned based on field duplicate results

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on WSMR groundwater sample MPL4-0112-1. Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample, MPL3-0112-2 was submitted for this sample delivery group (SDG). Field duplicate precision was acceptable and mercury was not detected at concentrations greater than the applicable LOQ.

### **VIII. Other**

A. LOQ were reviewed and found compliant

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on WSMR groundwater sample MPL4-0112-1. Matrix spike percent recoveries were outside, less than, laboratory acceptance windows of 90-100 percent for chloride and fluoride. Chloride results for all samples in this SDG are qualified as estimated results with J- for low bias. Fluoride results in all samples were non-detect and are qualified with the UJ flag. Percent recovery for sulfate was acceptable and RPD for all analytes was compliant.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A laboratory duplicate was analyzed and the results showed precision in compliance. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was compliant and RPD was calculated for elements when both sample and duplicate sample results were greater than the LOQ.

## **VIII. Other**

A. LOQs were reviewed for each sample. Fluoride LOQ was 2- to 4-times higher than normal and 4-times diluted sample analyses were reported non-detect. All samples were analyzed at dilution in order to get the chloride concentrations within calibration range and to protect the column and detector from high chloride concentrations. Fluoride and chloride results are qualified UJ and J-.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the laboratory. Field measurements were approximately 1 standard unit greater than laboratory measurements. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was compliant and RPD was calculated.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was compliant and RPD was calculated. A LCS duplicate was also analyzed and precision was found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was compliant and RPD was calculated.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **CONDUCTIVITY (Method 120.1)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was compliant and RPD was calculated.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **PHENOLS (Method 420.1)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was compliant and the analyte was not detected in either sample.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was compliant and the analyte was not detected in either sample.

### **VII. Other**

A. LOQ was reviewed and found compliant

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was compliant and the RPD was calculated for the reported results.

### **VII. Other**

A. LOQ was reviewed and found compliant. MS/MSD analyzed on WSMR groundwater sample MPL4-0112-1 was unusable. The sample concentration exceeded the spike level by more than 5-times.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was compliant and the RPD was calculated for the reported results. A LCS duplicate was also analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant. MS/MSD analyzed on sample MPL4-0112-1 showed compliant results.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was compliant and the RPD was calculated for the reported results. A LCS duplicate was also analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant. MS/MSD analyzed on sample MPL4-0112-1 showed compliant results.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field duplicate precision was compliant and the RPD was calculated for the reported results. A LCS duplicate was also analyzed and results found to be in compliance.

### **VII. Other**

A. LOQ was reviewed and found compliant. MS/MSD analyzed on sample MPL4-0112-1 showed compliant results.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping coolers temperatures were measured between 1°C and 3°C upon receipt at the laboratory. Sample temperatures were in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A field duplicate sample, MPL3-0112-2, was submitted for this SDG. Field precision could not be calculated as the original sample showed 13 mg/L suspended solids while the duplicate was non-detect above 5.0 mg/L. A LCS duplicate was also analyzed and results found to be in compliance. Data qualifiers were not assigned based on field precision only.

### **VI. Other**

A. LOQ was reviewed and found compliant. MS/MSD analyzed on sample MPL4-0112-1 showed compliant results.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, Main Post Landfill Wells MPL-10 through MPL-04, SDG L12020016, five samples, four field samples MPL1-0112-1, MPL2-0112-1, MPL3-0112-1, MPL4-0112-1, and one field duplicate MPL3-0112-2. Extra sample volume was collected at MPL4 for MS/MSD.**

### **Organic Compounds; VOC, PAH, and PCB – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Anions – Data Qualification Summary**

Chloride results in all samples were qualified as estimated values with low bias, J- flag. Fluoride results were flagged as estimated, J, in MPL1-0112-1, and as estimated, non-detect, UJ flag, in all other samples. Due to high chloride content samples were analyzed at 2- or 4-times dilution. Dilution raised the LOQ for fluoride. Matrix spike recoveries were less than criteria for chloride and fluoride.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Phenolics – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## OVERALL ASSESSMENT OF DATA

### **I. Compliance with method and project requirements**

A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

A. Based on the quality control criteria reviewed, all unqualified and qualified data are usable for project purposes. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” and “UJ” qualifiers. Estimated results are usable for limited purposes.

### **III. Field Duplicate Precision**

A. Field duplicate precision for the paired samples from MPL3 well are shown in the table below. RPD is only calculated when both sample results exceed the LOQ.

Method	Parameter	MPL3-0112-1		MPL3-0112-2		RPD
6010B	Aluminum	0.507		0.118		124.5%
6010B	Beryllium	0.00200	U	0.00200	U	NC
6010B	Boron	0.100	U	0.100	U	NC
6010B	Calcium	77.4		77.5		0.1%
6010B	Iron	0.592		0.298		66.1%
6010B	Magnesium	14.3		14.2		0.7%
6010B	Molybdenum	0.0100	U	0.0100	U	NC
6010B	Potassium	2.94		2.86		2.8%
6010B	Sodium	34.6		34.7		0.3%
6010B	Tin	0.500	U	0.500	U	NC
6010B	Vanadium	0.0100	U	0.0100	U	NC
6010B	Zinc	0.0547		0.0216		86.8%
6020	Antimony	0.00100	U	0.00100	U	NC
6020	Arsenic	0.00149		0.00137		8.4%
6020	Barium	0.0517		0.0503		2.7%
6020	Cadmium	0.000600	U	0.000600	U	NC
6020	Chromium	0.00235		0.00195	J	NC
6020	Cobalt	0.00100	U	0.00100	U	NC
6020	Copper	0.00200	U	0.00200	U	NC
6020	Lead	0.000829	J	0.00100	U	NC
6020	Manganese	0.0150		0.00396		116.5%
6020	Nickel	0.00381	J	0.00317	J	NC
6020	Selenium	0.00568		0.00514		10.0%
6020	Silver	0.00100	U	0.00100	U	NC

6020	Thallium	0.000200	U	0.000200	U	NC
7470A	Mercury	0.000200	U	0.000200	U	NC
300.0	Chloride	57.9		58.2		0.5%
300.0	Fluoride	0.800	U	0.800	U	NC
300.0	Sulfate	111		110		0.9%
9040	pH	7.47		7.40		0.9%
310.2	Alkalinity, total	122		129		5.6%
9014	Cyanide	0.234		0.282		18.6%
120.1	Conductivity	679		675		0.6%
420.1	Phenols	0.00556	U	0.00556	U	NC
350.1	Ammonia, as N	0.100	U	0.100	U	NC
353.2	Nitrate-Nitrite, as N	7.82		8.01		2.4%
SM4500-P-E-20th	Orthophosphate	0.132		0.139		5.2%
160.1	Total Dissolved Solids	408		366		10.9%
415.1	Total Organic Carbon	3.36		3.18		5.5%
160.2	Total Suspended Solids	13.0		5.0	U	NC



**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12020044**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: March 12, 2012

Matrix: Groundwater, 3 samples, 2 field samples and 1 field duplicate

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L120020044

Sample Nos.: MPL19-0212-1, MPL20-0212-1, and MPL20-0212-2

Comments: Field QC sample MPL20-0212-2 is a field QC duplicate of MPL20-0212-1 submitted for this SDG. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12020044  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	✓
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0	✓	✓	✓	✓	NA	✓	✓	NA	F 4X
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	NA
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	NA	✓	✓	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	NA	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on sample noted as internal laboratory QC (not a WSMR groundwater sample). Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance with one exception. Calcium percent recoveries (70.2 %R and 129 %R) exceeded both the lower and upper control limits. The parent sample concentration exceeded the spike level by greater than 4-times. Data validation qualifiers were not assigned.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this sample delivery group (SDG). Field duplicate precision was found to be in compliance.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

**IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant. Post digest spike results were in compliance for calcium.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in compliance with one exception. Cadmium and thallium were reanalyzed due to poor results on a low-level calibration check. The reanalysis was compliant.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance. The reanalysis was compliant.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample identified as internal laboratory QC (not a WSMR groundwater sample). All analyte recoveries and duplicate relative percent differences were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Field duplicate precision was found to be in compliance.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant. Post digest spike results were compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recovery and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. The parameter was not detected in either sample. MSD precision was found to be in compliance.

### **VIII. Other**

A. LOQ were reviewed and found compliant.

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were not reported for this SDG.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Duplicate precision was found to be compliant.

### **VIII. Other**

A. LOQ were reviewed and found compliant. Chloride concentrations in samples MPL20-0212-1 and the field duplicate were determined from 4-times dilution analysis.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show good agreement with 1 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Duplicate precision was found to be compliant.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Duplicate precision was found to be compliant.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Duplicate precision was found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **CONDUCTIVITY (Method 120.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Duplicate precision was found to be compliant.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **AMMONIA AS NITROGEN (Method 350.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Duplicate precision was found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Duplicate precision was found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Duplicate precision was found to be compliant. The parameter was not detected in either sample. A LCS duplicate was analyzed and the results for precision were compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Duplicate precision was 36.1% relative percent difference. A LCS duplicate was analyzed and the results were compliant. Data validation qualifiers were not assigned.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Duplicate precision was 15.4% relative percent difference. A LCS duplicate was analyzed and the results were compliant. Data validation qualifiers were not assigned.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. The field duplicate sample MPL20-0212-2 was submitted for this SDG. Duplicate precision could not be calculated as the parent sample result was 8.0 mg/L while the field duplicate was not detected above 5.0 mg/L reporting limit. A LCS duplicate was analyzed and the results were compliant. Data validation qualifiers were not assigned.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12020044, three samples; MPL19-0212-1. MPL20-0212-1, and field duplicate MPL20-0212-2.**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG. Chloride (300.0) result is from a 4-times diluted analysis. All other anions are reported from the undiluted analysis.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

- A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

- A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.

### **III. Field precision from the parent sample and field duplicate are summarized below:**

Method	Parameter	MPL20-0212-1		MPL20-0212-2		RPD
6010B	Aluminum	0.100	U	0.100	U	NC
6010B	Beryllium	0.00200	U	0.00200	U	NC
6010B	Boron	0.100	U	0.100	U	NC
6010B	Calcium	74.3		73.9		0.5%
6010B	Iron	0.187		0.178		4.9%
6010B	Magnesium	13.1		12.9		1.5%
6010B	Molybdenum	0.0100	U	0.0100	U	NC
6010B	Potassium	2.84		2.78		2.1%
6010B	Sodium	35.1		34.8		0.9%
6010B	Tin	0.500	U	0.500	U	NC
6010B	Vanadium	0.0100	U	0.0100	U	NC
6010B	Zinc	0.0260		0.0197	J	NC
6020	Antimony	0.00100	U	0.00100	U	NC
6020	Arsenic	0.00145		0.00131		10.1%
6020	Barium	0.0734		0.0741		0.9%
6020	Cadmium	0.000600	U	0.000600	U	NC
6020	Chromium	0.00172	J	0.00147	J	NC
6020	Cobalt	0.00100	J	0.00100	U	NC
6020	Copper	0.00200	U	0.01070		NC
6020	Lead	0.001000	U	0.001000	U	NC
6020	Manganese	0.0020	U	0.0020	U	NC
6020	Nickel	0.00253	J	0.00276	J	NC
6020	Selenium	0.00502		0.00451		10.7%
6020	Silver	0.00100	U	0.00100	U	NC
6020	Thallium	0.000200	U	0.000200	U	NC

7470A	Mercury	0.000200	U	0.000200	U	NC
300.0	Chloride	76.3		79.6		4.2%
300.0	Fluoride	0.800	U	0.800	U	NC
300.0	Sulfate	101		104		2.9%
9040	pH	7.39		7.45		0.8%
310.2	Alkalinity, total	89.6		91.6		2.2%
9014	Cyanide	0.242		0.222		8.6%
120.1	Conductivity	668		661		1.1%
350.1	Ammonia, as N	0.100	U	0.100	U	NC
353.2	Nitrate-Nitrite, as N	8.70		8.35		4.1%
SM4500-P-E-20th	Orthophosphate	0.050	U	0.050	U	NC
160.1	Total Dissolved Solids	304		438		36.1%
415.1	Total Organic Carbon	1.26		1.08		15.4%
160.2	Total Suspended Solids	8.0		5.0	U	NC



**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12020091**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: March 13, 2012

Matrix: Groundwater, 1 sample

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L12020091

Sample Nos.: MPL23-0212-1

Comments: Field QC samples were not submitted for this SDG. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12020091  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	✓
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0	✓	✓	✓	✓	✓	✓	✓	NA	✓
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	NA
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	NA	✓	✓	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	NA	✓	NA	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	NA	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on sample noted as internal laboratory QC (not a WSMR groundwater sample). Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample was not submitted for this sample delivery group (SDG). MSD precision was found to be in compliance.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

**IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant. Post digest spike results were compliant.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in compliance with exceptions for thallium noted in the report narrative. Thallium was reanalyzed due to poor results on a low-level calibration check. The reanalysis was compliant.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample identified as internal laboratory QC (not a WSMR groundwater sample). MS percent recoveries and duplicate relative percent differences were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recovery and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Other**

A. LOQ were reviewed and found compliant.

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recovery and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A laboratory duplicate was analyzed and the precision results were compliant.

### **VIII. Other**

A. LOQ were reviewed and found compliant.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show good agreement with 0.4 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A laboratory duplicate was analyzed on a non-project sample. Duplicate sample precision results were reviewed and found to be in compliance.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. Sample duplicates and LCS duplicates were run as evidenced on the run log. Precision data were not reported in the SDG. Data validation qualifiers were not assigned.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12020091, one sample MPL23-0212-1.**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

- A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

- A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.

COC No. A 28495

158 Starlite Drive  
Marietta, OH 45750

Microbac

Phone: 740-373-4071  
Fax: 740-373-4835



CHAIN-OF-CUSTODY RECORD

Company Name: <b>Zia / SHAW</b>						NUMBER OF CONTAINERS	Hold	Anions	Cond pH PO4	Alkalinity	Total, Free, Amenable CN	Metals	NH3 TOC NO3NO2	TSS TDS	TOTAL # (LAB USE)	Program		
Project Contact: <b>Mark Lyon</b>		Contact Phone #: <b>505-262-8920</b>		<input type="checkbox"/> CWA	<input type="checkbox"/> RCRA													
Turn Around Requirements: <b>Normal</b>		Location: <b>WSMR</b>		<input type="checkbox"/> DOD	<input type="checkbox"/> AFCEE													
Project ID: <b>STP</b>		Sampler (print): <b>Bradley T. Davis</b> <b>BrT-Davis</b>		Signature: <b>BrT-Davis</b>														
Sample I.D. No.	Comp	Grab	Date	Time	Matrix*												ADDITIONAL REQUIREMENTS	
<b>MPL 24-0212-1</b>		<b>X</b>	<b>2-3-12</b>	<b>1030</b>	<b>W</b>	<b>7</b>												
Relinquished by: (Signature) <b>BrT-Davis</b>			Date: <b>2-3-12</b>	Time: <b>1700</b>	R (€)	Microbac OVD		Received: 02/04/2012 11:21		By: BOB BUCHANAN		221000022134		Date	Time	Received by: (Signature)		
Relinquished by: (Signature)			Date	Time	R (€)	<b>RF Buchanan</b>								Remarks:				

\*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12020146**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: March 13, 2012

Matrix: Groundwater, 1 sample

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L12020146

Sample Nos.: MPL24-0212-1

Comments: Field QC samples were not submitted for this SDG. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

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- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

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- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

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CCB	Continuing calibration blank contamination
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PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12020146  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	Ca 10x
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0	✓	✓	✓	✓	NA	✓	✓	NA	2x
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	NA
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	NA	✓	NA	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	NA	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on sample noted as internal laboratory QC (not a WSMR groundwater sample). Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance except for aluminum and iron. The MS/MSD analyses were repeated and calcium and iron again were non-compliant. Calcium concentration in the parent sample was greater than 4-times the spike concentration. As the MS/MSD parent sample was not identified as a WSMR sample, data validation qualifiers were not assigned.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample was not submitted for this sample delivery group (SDG). MSD precision was in compliance except as noted for aluminum and iron. Validation qualifiers were not assigned.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

## **IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant. Post-digest spike recovery results were compliant. Calcium was analyzed at a 10-times dilution in order to obtain a result within the calibration range.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance with one exception. Thallium was reanalyzed due to thallium detected in a continuing calibration blank. The reanalysis was compliant.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample identified as internal laboratory QC (not a WSMR groundwater sample). MS percent recoveries and duplicate relative percent differences were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A laboratory duplicate sample was analyzed in this SDG. Laboratory duplicate and MSD precision were compliant.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant. Post-digest spike recoveries were compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recovery and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Other**

A. LOQ were reviewed and found compliant. The post-digest spike results were compliant.

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were not reported for this SDG.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A laboratory duplicate was analyzed and the precision results were compliant.

### **VIII. Other**

A. LOQ were reviewed and found compliant. Chloride, fluoride, and sulfate concentrations were determined from a 25-times dilution analysis.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show good agreement with 0.5 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A laboratory duplicate was analyzed. Duplicate sample precision results were reviewed and found to be in compliance.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. A laboratory duplicate was analyzed as evidenced on the instrument run log. The duplicate analysis was not on a WSMR sample. Duplicate precision was not reported in this SDG.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 1 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12020146, one sample MPL24-0212-1.**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG. Calcium (6010B) result is from a 10-times diluted analysis.

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG. Chloride, fluoride, and sulfate (300.0) results are from a 2-times diluted analysis.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.

COC No. A 28508

158 Starlite Drive  
Marietta, OH 45750

Microbac

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Fax: 740-373-4835

CHAIN-OF-CUSTODY RECORD

Company Name: <b>Zia / SHAW</b>						NUMBER OF CONTAINERS	Hold	Anions	Cond pH	PO4	Alkalinity	Total, Free, Ammonia CN	Metals	NH3 TOC NO3NO2	TSS TDS	TOTAL # (LAB USE)	Program	
Project Contact: <b>Mark Lyon</b>			Contact Phone #: <b>505-262-8920</b>														<input type="checkbox"/> CWA	<input type="checkbox"/> RCRA
Turn Around Requirements: <b>Normal</b>			Location: <b>WSMR</b>														<input type="checkbox"/> DOD	<input type="checkbox"/> AFCEE
Project ID: <b>STP</b>			Signature: <b>Bradley T. Davis</b>														<input type="checkbox"/> Other	
Sampler (print): <b>Bradley T. Davis</b>			Signature: <b>Bradley T. Davis</b>			ADDITIONAL REQUIREMENTS												
Sample I.D. No.	Comp	Grab	Date	Time	Matrix*													
MPL25-0212-1		X	2-6-12	1505	W	7		X	X	X	X	X	X	X				
SMW1-0212-1		X	2-7-12	0950	W	7		X	X	X	X	X	X	X				
SMW4-0212-1		X	2-7-12	1205	W	7		X	X	X	X	X	X	X				
Relinquished by: <b>Bradley T. Davis</b>						Date: <b>2-7-12</b>	Time: <b>1700</b>	Received by: <b>Erin Porter</b>		Received by: <b>Erin Porter</b>		Date: _____		Time: _____		Received by: _____		
Relinquished by: _____						Date: _____	Time: _____	Received by: _____		Received by: _____		Date: _____		Time: _____		Received by: _____		
Remarks: _____																		

\*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L12020220**

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-62, Former STP Percolation Ditches

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc.

Review Date: February 8, 2012

Matrix: Groundwater, 3 samples

Parameters: Metals 6010B/6020/7470A  
Anions 300.0  
pH 9040C  
Alkalinity 310.2  
Cyanide 9014/9010C/SM4500-CN-C, G/SM4500-CN-I  
Conductivity 120.1  
Ammonia-Nitrogen 350.1  
Nitrate+Nitrite-Nitrogen 353.2  
Orthophosphate SM4500-P-E-20th  
Total Dissolved Solids 160.1/SM2540C  
Total Organic Carbon 415.1  
Total Suspended Solids 160.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L12020220

Sample Nos.: MPL25-021201, SWM1-0212-1, SWMU4-0212-1

Comments: Field QC samples were not submitted for this SDG. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

## DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (\*).

### Organic Parameters

- \* Temperature
- \* Holding times
- GC/MS instrument performance check
- \* Initial and continuing calibrations
- \* Blanks
- \* Surrogate recoveries
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- \* Overall data assessment

### Inorganic and General Chemistry Parameters

- \* Temperature
- \* Holding times
- \* Initial and continuing calibration
- \* Blanks
- \* Matrix spike/matrix spike duplicate
- \* Laboratory control sample / blank spike
- \* Field duplicate
- \* Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- \* ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- \* Overall data assessment

## DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

## DATA VALIDATION QUALIFIER REASON CODES

Reason Code	Data Quality Condition Resulting in Assigned Qualification
<b>General Use</b>	
FB	Field blank contamination
FD	Field duplicate evaluation criteria not met
HT	Holding time requirement was not met
PR	Preservation requirements not met
LCS	Laboratory control sample evaluation criteria not met
MB	Method blank or preparation blank contamination
RB	Rinsate blank contamination
TB	Trip blank contamination
SDL	Sample quantitation limit exceeds decision criteria and the analyte was not detected
<b>Inorganic Methods</b>	
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification evaluation criteria not met
D	Laboratory duplicate precision evaluation criteria not met
DL	Serial dilution results did not meet evaluation criteria
ICS	Interference check sample evaluation criteria not met
ICV	Initial calibration verification evaluation criteria not met
MS	Matrix spike recovery outside acceptance range
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
PB	Preparation blank
<b>Organic Methods</b>	
CCAL	Continuing calibration evaluation criteria not met
ICAL	Initial calibration evaluation criteria not met
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
MS/MSD	Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met
SUR	Surrogate recovery outside acceptance range
TUNE	Instrument performance (tuning) criteria not met
P	The detected concentration difference between the primary and secondary column is greater than 25%.

**SAMPLE DELIVERY GROUP L12020220  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	✓	✓
Metals 6020	✓	✓	✓	✓	✓	✓	✓	✓	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0	✓	✓	✓	✓	✓	✓	✓	NA	Cl
pH 9040C	✓	✓	✓	NA	NA	✓	✓	NA	NA
Alkalinity 310.2/SM2320B	✓	✓	✓	✓	NA	✓	✓	NA	NA
Cyanide; Total, Amenable, Dissociable 9014-9010C/SM4500-CN	✓	✓	✓	✓	NA	✓	✓	NA	✓
Conductivity 120.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Ammonia-N 350.1	✓	✓	✓	✓	NA	✓	NA	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	NA	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	NA	✓	✓	NA	✓
Total Organic Carbon 415.1	✓	✓	✓	✓	NA	✓	✓	NA	✓
Total Suspended Solids 160.2	✓	✓	NA	✓	NA	✓	✓	NA	✓

**Notes:**

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

**DATA ASSESSMENT**  
**METALS (Method 6010B)**

**I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

**II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

**III. Calibration**

A. Calibration linearity, initial calibration verification (ICV), and continuing calibration verification (CCV), were reviewed and found to be in compliance.

**IV. Blanks**

A. Initial calibration blank (ICB), continuing calibration blank (CCB), and method blank analysis results were reviewed and found to be in compliance.

**V. Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted as internal laboratory QC (not a WSMR groundwater sample). Matrix spike percent recoveries and duplicate relative percent difference were found to be in compliance with one exception. Calcium percent recovery (126 %R) was greater than the upper control limit. Calcium concentration in the parent sample was greater than 4-times the matrix spike. Data validation qualifiers were not assigned.

**VI. Laboratory Control Sample (LCS)**

A. The LCS results were reviewed and found to be in compliance.

**VII. Duplicate**

A. A field duplicate sample was not submitted for this sample delivery group (SDG). MSD precision was found to be in compliance.

**VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

**IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant. Post-digest spike percent recoveries were compliant.

## **METALS (Method 6020)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, low-level calibration check, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample identified as internal laboratory QC (not a WSMR groundwater sample). MS percent recoveries and duplicate relative percent differences were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Serial Dilution**

A. Serial dilution results were reviewed and found to be in compliance.

### **IX. Other**

A. LOQ were reviewed and found compliant. Post digest spike results were compliant.

## **MERCURY (Method 7470A)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration linearity, ICV, and CCV were reviewed and found to be in compliance.

### **IV. Blanks**

A. ICB, CCB, and method blank analysis results were reviewed and found to be in compliance. As narrated by the laboratory, a CCB did show mercury contamination greater than the LOD and less than the LOQ. However, mercury was not detected in the associated field samples above the LOD. Validation qualifiers were not assigned.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not a WSMR groundwater sample). MS/MSD percent recovery and duplicate relative percent difference were found to be in compliance.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision was found to be in compliance.

### **VIII. Other**

A. LOQ were reviewed and found compliant. The post digest spike results were compliant.

## **ANIONS (Method 300.0 [9056])**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank and CCB analysis results were reviewed and found to be in compliance.

### **V. MS/MSD**

A. MS/MSD analyses were performed in the applicable analytical batch on a sample noted to be internal laboratory QC (not necessarily a WSMR groundwater sample). MS/MSD percent recoveries and precision measures were compliant except for chloride which recovered less than the lower acceptance limit. Chloride concentration in the parent sample exceeded the spike amount by greater than 4-times. Data validation qualifiers were not assigned.

### **VI. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VII. Duplicate**

A. The MSD was analyzed and precision measures were compliant.

### **VIII. Other**

A. LOQ were reviewed and found compliant. Chloride concentration was determined from a 5-times dilution analysis of sample MPL25-0212-1 and from a 2-times dilution analysis of sample SMW4-0212-1 in order to get results within the calibration curve.

## **pH (Method 9040C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. Hydrogen ion activities (pH) were measured in the field at the time of sample collection and again at the. The paired measurements show good agreement with 0.5 standard units difference. Data validation qualifiers were not assigned.

### **III. Calibration**

A. pH meter calibration at the laboratory was documented using buffer solutions pH 4, 7, and 10.

### **IV. LCS**

A. The LCS sample pH was measured at the laboratory and the results were acceptable.

### **V. Duplicate**

A. A laboratory duplicate was analyzed on a non-project sample. Duplicate sample precision results were reviewed and found to be in compliance.

## **ALKALINITY (Methods 310.2 / SM2320B)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

### **IV. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

## **CYANIDE (Methods 9014-9010C / SM4500-CN-C)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blanks were reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance. Cyanide amenable to chlorination is a difference measurement which the LCS calculation accounts for inversely.

### **VI. Duplicate**

A. A laboratory duplicate sample analysis was evidenced on the instrument run log. Duplicate precision appeared compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## CONDUCTIVITY (Method 120.1)

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## AMMONIA AS NITROGEN (Method 350.1)

### **I. Temperature**

- A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

- A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

- A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

- A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

- A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

- A. A laboratory duplicate was evidenced on the instrument run log. Duplicate analysis results were not reported in this SDG.

### **VII. Other**

- A. LOQ was reviewed and found compliant.

## **NITRATE + NITRITE AS NITROGEN (Method 353.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **ORTHOPHOSPHATE AS PHOSPHOROUS (Method SM4500-P)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation was reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL DISSOLVED SOLIDS (Method 160.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL ORGANIC CARBON (Method 415.1)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Calibration**

A. Calibration coefficient of correlation and ICV were reviewed and found compliant.

### **IV. Blanks**

A. Method blank was reviewed and found to be in compliance.

### **V. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **VI. Duplicate**

A. A LCS duplicate was analyzed. The results were reviewed and found to be compliant.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **TOTAL SUSPENDED SOLIDS (Method 160.2)**

### **I. Temperature**

A. Shipping cooler temperature was measured at 0 °C upon receipt at the laboratory. Sample temperature was in compliance.

### **II. Holding Times**

A. The analysis holding times were reviewed and found to be in compliance.

### **III. Blanks**

A. Method blank results were reviewed and found to be in compliance.

### **IV. LCS**

A. The LCS results were reviewed and found to be in compliance.

### **V. Duplicate**

A. A LCS duplicate was analyzed and results found to be in compliance.

### **VI. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L12020220, three samples MPL25-0212-1, SMW1-0212-1, and SWM4-0212-1.**

### **Metals – Data Qualification Summary**

No sample data were qualified in this SDG.

### **Anions – Data Qualification Summary**

No sample data were qualified in this SDG. Chloride (300.0) results were from diluted analyses as appropriate. All other anions are reported from the undiluted analysis.

### **Cyanide – Data Qualification Summary**

No sample data were qualified in this SDG.

### **General and Wet Chemistry – Data Qualification Summary**

No sample data were qualified in this SDG.

## **OVERALL ASSESSMENT OF DATA**

### **I. Compliance with method and project requirements**

- A. All analyses were performed within the analytical methods specifications and project requirements.

### **II. Usability**

- A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.