

**DATA VALIDATION REPORT**  
**MICROBAC LABORATORY SDG L13110584**

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. a CB&I Company

Review Date: December 10, 2013

Matrix: Groundwater, one field sample with extra volume for MS/MSD

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: L13110584

Sample Nos.: MPL21-1113-1

Comments: Additional volume submitted for MS/MSD

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 L13110584  
LEVEL III DATA VALIDATION SUMMARY**

Analysis / Method	Temperature	Holding Times	Calibration	Blanks	MS/MSD	LCS	Duplicate	Serial Dilution	Other
Metals 6010B	✓	✓	✓	✓	✓	✓	✓	NA	✓
Metals 6020	✓	✓	✓	✓	✓	✓	✓	NA	✓
Mercury 7470A	✓	✓	✓	✓	✓	✓	✓	NA	✓
Anions 300.0	✓	✓	✓	✓	✓	✓	✓	NA	✓
pH 9040C	✓	✓	✓	NA	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	✓	✓	✓	<b>15</b>	<b>15</b>	✓	✓	NA	✓
Nitrate+Nitrite-N 353.2	✓	✓	✓	✓	✓	✓	✓	NA	✓
Orthophosphate SM4500-P	✓	✓	✓	✓	✓	✓	✓	NA	✓
Total Dissolved Solids 160.1	✓	✓	NA	✓	✓	✓	✓	NA	NA
Total Organic Carbon 415.1	✓	✓	✓	✓	✓	✓	✓	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 with exception. Method blank contained zinc at a concentration less than ½ the LOQ. The field sample result was non-detect. Validation qualifiers were not assigned.

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

A. MS/MSD analyses were performed on sample MPL21-1113-1. MS/MSD recoveries met acceptance criteria.

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

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

**VII. Duplicate**

A. Field duplicate samples were not submitted for this SDG. MSD precision met acceptance criteria.

**VIII. Serial Dilution**

A. Serial dilution was performed on a non-WSMR sample in the analytical batch and was not evaluated.

## **IX. Other**

A. Limits of Quantitation (LOQ) were reviewed and found compliant. Post digest spike analysis was performed on a non-WSMR sample in the analytical batch and not evaluated.

## **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 on sample MPL21-1113-1. MS/MSD results met acceptance criteria.

### **VI. LCS**

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

### **VII. Duplicate**

A. Field duplicate samples were not submitted for this SDG. MSD precision met acceptance criteria.

### **VIII. Serial Dilution**

A. Serial dilution was performed on a non-WSMR sample and not evaluated.

### **IX. Other**

A. LOQ were reviewed and found compliant. Post digest spike analysis was performed on a non-WSMR sample and not evaluated.

## **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 on sample MPL21-1113-1. MS/MSD results met acceptance criteria.

### **VI. LCS**

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

### **VII. Duplicate**

A. Field duplicate samples were not submitted for this SDG. MSD precision met acceptance criteria.

### **VIII. Other**

A. LOQ were reviewed and found compliant. Post digest spike was performed on a non-WSMR sample and was not evaluated.

## **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 were performed on sample MPL21-1113-1. Percent recoveries met acceptance criteria with exceptions. Fluoride recovered at 88.4 percent and 89.2 percent which are less than the lower laboratory limit of 90 percent. Validation qualifiers were not assigned. Recoveries for chloride and sulfate met acceptance limits.

### **VI. LCS**

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

### **VII. Duplicate**

A. Field duplicate samples were not submitted for this SDG. MSD precision met acceptance criteria.

### **VIII. Other**

A. LOQs met project requirements. The sample was analyzed undiluted.

## **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 laboratory.

### **III. Calibration**

A. pH meter calibration at the laboratory was checked using buffer solutions pH 7.0 and 10.0.

### **IV. LCS**

A. The LCS sample pH was indicated on the instrument run log.

### **V. Duplicate**

A. Field duplicate samples were not submitted for this SDG. A laboratory duplicate was run on sample MPL21-1113-1 as indicated on the run log. Precision was acceptable.

## **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. MS/MSD**

A. MS/MSD were performed on MPL21-1113.1. Percent recoveries were less than the laboratory acceptance limits, however the titration analyses does not lend itself to spike testing. Validation qualifiers were not assigned.

### **VI. LCS**

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

### **VII. Duplicate**

A. Field duplicate samples were not submitted for this SDG. An LCS duplicate and the MSD demonstrated acceptable precision.

## **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. MS/MSD**

A. MS/MSD were performed on MPL21-1113-1. Percent recoveries met 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 field duplicate sample was not submitted for this SDG. MSD precision met acceptance criteria.

### **VIII. 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. Conductivity detected in the method blank at less than ½ the LOQ. Validation qualifiers were not qualified.

### **IV. LCS**

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

### **V. Duplicate**

A. A field duplicate sample was not submitted for this SDG.

### **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. The preparation blank, ICB, and CCB all showed negative results the absolute value of which exceeded the LOQ. Sample results were non-detect and are qualified "UN" for estimated non-detect with reason codes "PB, ICB, and CCB."

### **V. LCS**

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

### **VI. MS/MSD**

A. MS/MSD was performed on MPL21-1113-1. Percent recoveries were less than the lower acceptance limit but greater than 40 percent. Non-detect results are previously qualified "UN" as estimated non-detect and reason code "MS" is added to qualification.

### **VII. Duplicate**

A. A field duplicate sample was not submitted for this SDG. MSD precision met acceptance criteria.

### **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 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.

### **VI. MS/MSD**

A. MS/MSD was analyzed on sample MPL21-1113-1. Percent recoveries were 79 percent and 112 percent, respectively, which are outside the laboratory acceptance limits of 90 percent to 110 percent. All other quality control measures met specifications. 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. Precision for lab control sample duplicate and MSD met acceptance criteria.

### **VIII. 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. MS/MSD**

A. MS/MSD was performed on sample MPL21-1113-1. Percent recoveries met acceptance limits.

### **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. Precision for LCS duplicate and MSD were 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 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. The method blank reported a negative result the absolute value of which was greater than ½ the LOQ. Field sample results were orders of magnitude greater than the blank values. Data validation qualifiers were not assigned.

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

A. MS/MSD were performed on sample MPL21-1113-1. Percent recoveries met acceptance limits.

### **V. LCS**

A. The LCS results were reviewed and met acceptance criteria.

### **VI. Duplicate**

A. A field duplicate sample was not submitted for this SDG. Precision results for the LCS duplicate and MSD were 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 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. MS/MSD**

A. MS/MSD were performed on sample MPL21-1113-1. Percent recoveries met acceptance criteria.

### **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. LCS duplicate and MSD precision results met acceptance criteria.

### **VIII. 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. MS/MSD**

A. MS/MSD were performed on sample MPL21-1113-1. Percent recoveries met acceptance criteria.

### **V. LCS**

A. The LCS results recovered within the acceptance limits.

### **VI. Duplicate**

A. A field duplicate sample was not submitted for this SDG. LCS duplicate and MSD precision measurement met acceptance criteria.

### **VII. Other**

A. LOQ was reviewed and found compliant.

## **DATA QUALIFICATION SUMMARY**

**CCWS-62, Former STP Percolation Ditches, SDG L13110584; one field sample with extra volume for MS/MSD:**

MPL21-1113-1.

### **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**

For ammonia as nitrogen, the preparation blank, ICB, and CCB all showed negative results the absolute value of which exceeded the LOQ. Sample results were non-detect and are qualified “UN” for estimated non-detect with reason codes “PB, ICB, and CCB.” In the MS/MSD percent recoveries were less than the lower acceptance limit but greater than 40 percent. Non-detect results are previously qualified “UN” as estimated non-detect and reason code “MS” is added to qualification.

## **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 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.