Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

Project No: **Eco-18-1237**

SDG #23J004 Analytical Data Package

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By
ZIBA HOSSEINI

14343 Peach Hill Rd.
Moorpark, CA, 93021

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2023 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of nine (9) water samples were collected on 10-02-23. EMAX Laboratories received the samples on 10-03-23. Data was delivered in one package as stage 2b and stage 3 deliverable. Data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples was submitted for the requested analytical method. Sample TMW44102023 (Lab ID#J004-01) was selected to be reviewed as stage 3 deliverable. Raw data for this sample was compared to the reported summary tables and went through comprehensive data validation. No sample was designated to be spiked as MS/MSD. Recoveries and results of LCS/LCSD was used to evaluate both accuracy and precision. Raw data for method blank and LCS/LCSD were cross checked with the corresponding summary table results.

Stage 2b data validation examined quality assurance/quality control (QA/QC) elements such as holding time (sampling to analysis), instrument injection logs, method blank results, QC summary results and recoveries, LODs/LOQs, summaries of initial and continuing calibrations and completeness of results for the following requested EPA method of analysis:

EPA Method 9056A: Anions by IC (9 samples)

The analytical results, QC results, initial calibration summary table and initial calibration verification (ICV) data were comprehensively compared with the corresponding raw data and chromatograms presented for stage 3 data validation.

All the requested samples were analyzed for each of the components listed in the corresponding EPA Method (QAPP; final version, Eco & Associate, Inc. April 2019). The evaluation indicated that all the analytical work was performed as requested on the chain of custody. The required analytical holding times were met for all anions. The deviations, if any, are discussed in Section 4.0 for this method.

The analytical data evaluated in this data validation report (SDG # 23J004) has met the data quality and usability requirement as defined in the data quality objectives. Overall data is of acceptable quality and considered usable for its intended purpose.

1.0 INTRODUCTION

This report presents the evaluation and validation of analytical data for water samples collected as a part of water monitoring at Fort Wingate, New Mexico (NM).

1.1 Objectives and Scope of Data

The main objective of this report is to assess the acceptability of the data generated by the designated laboratory. The data validation was performed according to the analytical requirements of the method in the *Quality Assurance Project Plan, final Draft, USACE Fort Wingate New Mexico,* (Project No: Eco-18-1237, April 2019), *USEPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Organic Data Review* (USEPA, January 2017), *National Functional Guidelines for Inorganic Data Review* (USEPA, September 2016), US DoD General Data Validation Guideline, February 2018, *EM 200-1-10 Guidance for Evaluating Performance-based Chemical Data, US Army Corps of Engineers (USACE). June 2005 and DoD Quality* System Manual, QSM 5.3, 2019. The approved site-specific Quality Assurance Project Plan (ECO QAPP) has the highest hierarchy.

1.2 Organization of the Report

Section 2.0 describes the components of the data review. Section 3.0 provides the qualitative quality assurance objectives. Section 4.0 summarizes the findings and conclusions of the data validation.

2.0 DATA REVIEW AND VALIDATION

Data validation is a systematic method for reviewing and qualifying the presented analytical data for their intended use. The objective of this data validation report is to identify any unacceptable or faulty measurements, as reported by the laboratory.

EMAX Laboratories in Torrance, California performed the chemical analysis of the samples. Army Corps of Engineers and the State of California has certified this laboratory for performing the analysis described within this report.

A total of nine (9) water samples were collected on 10-02-23. EMAX Laboratories received the samples on 10-03-23.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. Data was subjected to validation to the equivalent of stage 3.

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)
- Result and percent recoveries of MS/MSD, when applicable
- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;

- Case narrative for each method;
- Raw data for initial calibration, initial calibration verification, continuing calibrations, and chromatograms for the sample/samples at Stage 3 deliverable and related QC samples.

Data validation was performed by initial review of the analytical reports and QA/QC results and recoveries using summary tables. Then, selected analytical reports including QA/QC information was cross checked with raw data. The analysis sequence log for the method was examined. Overall review assessed the effects of QA/QC results on the data usability. The review included such parameters as holding times, LODs/LOQs, initial and continuing calibration method requirements, MS/MSD results and lab control sample (LCS) results and percent recoveries for accuracy and precision.

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for analytical method requested on the chain of custody were submitted for each sample. Sample TMW44102023 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample together with QC samples were evaluated comprehensively at stage 2b and stage 3 data validation review. No sample was spiked as MS/MSD. Raw data for LCS/LCSD was reviewed. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);
- Technical holding times were confirmed for all samples with reference to the requested method of analysis (collection to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;

- Continuing calibration standards were evaluated
- MS/MSD results, if requested, were evaluated;
- LCS/LCSD results were evaluated; and
- Method blank results as well as surrogate recoveries were evaluated.

The following is a list of field sample identification and corresponding laboratory sample identification number:

Site Name: Fort Wingate, New Mexico

SDG#23J004 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
TMW44102023	J004-01	10-02-23	S3VM	Anions by IC
MW23102023	J004-02	10-02-23	S3VM	Anions by IC
BGMW12102023	J004-03	10-02-23	S3VM	Anions by IC
TMW46102023	J004-04	10-02-23	S3VM	Anions by IC
MW32102023	J004-05	10-02-23	S3VM	Anions by IC
BGMW03102023	J004-06	10-02-23	S3VM	Anions by IC
TMW18102023	J004-07	10-02-23	S3VM	Anions by IC
BGMW08102023	J004-08	10-02-23	S3VM	Anions by IC
TMW36102023	J004-09	10-02-23	S3VM	Anions by IC

TABLE 2-1
Summary of Analytical Parameters
USACE Wingate, New Mexico

Table 2-1 below shows the specified analysis for constituents in the water samples, the corresponding Environmental Protection Agency (EPA) analytical method, and the corresponding limit of quantitation (LOQ), of groups of constituents.

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperature upon receipt in the Laboratory were reviewed. Samples were received in 1 cooler.

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, and analysis. Collection to analysis was within the holding time requirement.

Table 2-2 presents the summary of holding time requirement with qualifications if applied.

TABLE 2-2

Summary of Analytical Methods and Holding Time Requirements
USACE Wingate, New Mexico

ANALYSIS Method	MATRIX	HOLDING TIME REQUIREMENT	DATA QUALIFIED AS "J"
Anions by IC	Water	Analysis within 48 hours for Nitrate, Nitrite and Orthophosphate	None. Holding times were met for samples analyzed
		Analysis within 28 days for Bromide, Fluoride, Chloride and Sulfate	None. Holding times were met for samples analyzed

2.2.3 Laboratory and Field Blanks

The objective of laboratory and field blanks is to determine the presence and extent of contamination resulting from laboratory or field activities. Blanks reported here included method blank only. The result of analysis of method blank is discussed in Section 4.0 for this method. Samples were transported in one ice preserved cooler and was stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as 2.9˚C upon arrival. Samples were received in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

Quality assurance (QA) objectives define analytical parameters that validate the conclusions drawn from the results. Quality assurance was assessed through the following means: precision, accuracy, representativeness, completeness, and comparability (PARCC).

3.1 Qualitative QA Objectives

Qualitative aspects of QA for analytical data are characterized by completeness and representativeness.

3.1.1 Comparability

Comparability defines the level of confidence with which one data set can be compared with another. Comparability is related to accuracy and precision. It is also a measure of the data's reliability. All units for comparability are in accordance with standard procedures so that the results could be compared with other laboratories if necessary.

3.1.2 Representativeness

Representativeness is a quantity, which presents whether the results of analysis accurately portray the actual site conditions. Representativeness is a qualitative parameter, which signifies the extent of accuracy and precision, to which the data represent a characteristic population, parameter variations at a sampling point, process condition, or environmental conditions. The sampling procedures described within the approved QAPP (Eco & Associate, Inc., April 2019) are designed to provide samples representative of the site conditions.

3.2 Quantitative QA Objectives

Quantitative QA Objectives for analytical data are defined as precision, accuracy, completeness, and method quantitation limits. These quantitative parameters are established in order to monitor the overall quality of analytical data produced by the laboratory. The laboratory performing the analytical methods specified in Table 2-1, and the case narratives, which is included in the data package from the laboratory, ensures the quality of the analytical data.

3.2.1 Precision

Precision is a measure of the closeness with which multiple analyses of a given sample agree with each other. It describes the agreement between two or more measurements that have been made in similar condition. Precision is measured through matrix spike/matrix spike duplicate samples, laboratory control sample/ laboratory control sample duplicate and sample/sample duplicate analysis. In the latter case, the sample with positive results can be used for this purpose. The relative percent difference (RPD) is calculated as a means of quantifying precision. The following equation is used for this purpose:

$$RPD = \frac{R_1 - R_2}{(R_1 + R_2)/2} X 100$$

Where:

RPD = Relative percent difference

R_1 = Result of the first duplicate or measured sample concentration

R_2 = Result of the second duplicate or known sample or duplicate concentration

When analytes are present at concentrations below or near the quantitation limit, precision is measured, using MS/MSD, and/or LCS/LCSD results.
Precision results are discussed in Section 4.0 of this report.

3.2.2 Accuracy

Accuracy indicates the closeness of the measurement to its true or accepted value. Accuracy measures agreement between a result and its true value. Accuracy is measured through laboratory control sample analysis and surrogate recoveries. Method-specific QA objectives for precision and accuracy were based on the quality control limits developed by the laboratory for the analytical methods, specified in Table 2-1. These procedures may affect the accuracy of the data presented. Additionally, initial, and continuing calibrations were used to verify that the analytical instrument accurately measured the compound concentrations. Calculations were independently verified for the responses and percent differences (%Ds).

3.2.3 Completeness

Completeness is defined as the percentage of total measurements, which are judged to be valid. Completeness objective is to obtain sufficient measures of valid data to enable the goals and objectives of the project to be achieved.

Completeness is quantified by computing the fraction of reports, which remained valid after the sampling procedures were reviewed and the results conformed to QA/QC protocols. The following equation was used to calculate completeness:

$$\text{Completeness} = \frac{\text{No. of valid field samples collected and analyzed}}{\text{No. of valid field samples reported}} \text{ X } 100$$

Completeness (EPA Method 9056A: Anions) =9/9X100=100%

Completeness is affected by anything that reduces the number of samples analyzed (such as a sample loss during transport or extraction), as well as acceptance or non-acceptance of analytical results.

4.0 DATA VALIDATION

This data review covers nine water samples listed on page 8 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Method:

EPA Method 9056A for Bromide, Chloride, Fluoride, Nitrate and Nitrite,
Orthophosphate and Sulfate by IC

This review follows *Quality Assurance Project Plan, final Draft, USACE Fort Wingate Depot Activity*, McKinley County, New Mexico; Project # Eco-18-1237 April 2019, EM *200-1-10 Guidance for Evaluating Performance-based Chemical Data; US Army Corps of Engineers (USACE). June 2005,* and USEPA Analytical Operations/Data Quality Center (*AOC) National Functional Guidelines for Organic Data Review* (USEPA, January 2017); DoD QSM 5.3, 2019 and *National Functional Guidelines for Inorganic Data Review* (USEPA, September 2016). The Approved site-specific Quality Assurance Project Plan has the highest hierarchy.

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

U	Indicates the analyses was analyzed for but not detected at or above Limit of Detection (LOD).
J	Indicates an estimated value with an unknown bias.
UJ	Indicates the analyte was analyzed for but not detected and reported less than LOD. However, the numerical value is approximate.
J^+	The result was estimated value and may be biased high.
J^-	The result was estimated value and may be biased low.
X	The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality criteria. The presence or absence of the analyte cannot be confirmed by the data provided. Acceptance or rejection of the data should be decided by the project team, but exclusion of the data is recommended

The following Reason codes were applied in the report:

M3	MS/MSD and/or LCS/LCSD percent recovery infraction with low bias
M4	MS/MSD or duplicate precision infraction
S1	Surrogate percent recovery infraction with high bias
S2	Surrogate percent recovery infraction with low bias
R4	Result exceeds calibration range
B6	Trip blank infraction (qualified detect)
B7	Field blank infraction (qualified detect)
B8	Equipment blank infraction (qualified detect)
D1	Field duplicate precision infraction

4.1. Method SW9056A: Bromide, Fluoride, Chloride, Nitrate-N, Nitrite, Orthophosphate, and Sulfate

4.1.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of water samples requested for this method. A total of nine (9) water samples were collected on 10-02-23. Samples were analyzed on 10-03-23 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Samples were re-analyzed on 10-04-23 and 10-05-23 for Sulfate and Chloride within 28-day holding time.

4.1.2. Initial and continuing calibration: Anions such as Chloride, Fluoride, Bromide, Nitrite, Nitrate, Orthophosphate and Sulfate were separated from water samples by Ion chromatography. The separated anions in their acid form (very conductive) were measured by conductivity. They were identified based on retention time as compared to reference standards.

Instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 09-18-23. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each anion. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 09-19-23. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-03-23, 10-04-23 and 10-05-23. A total of nine continuing calibration standards were analyzed with analysis and re-analysis of all samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All method blanks were reported as non-detected for the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.1.3. Quality Control Samples consisted of one method blank and one set of LCS/LCSD. No sample was spiked as MS/MSD. A second set of LCS/LCSD were analyzed with re-analysis of Chloride and Sulfate on 10-04-23. Recoveries of LCS/LCSDs were within 90-110 % of spiked values for each anion. Percent RPDs were less than 20% for each set of LCS/LCSD for all anions.

4.1.4. Field duplicate sample and its associated sample: No field duplicate was collected with this sample delivery group.

4.1.5. Raw data was submitted all field samples analyzed. Sample TMW44102023 (Lab ID#23J004-01) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #23J004 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

6.0 REFERENCES

1. *USEPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Organic Data Review* (USEPA, January 2017).

2. *USEPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Inorganic Data Review* (USEPA, September 2016).

3. *Quality Assurance Project Plan, final Draft, USACE Fort Wingate, NM* (Project No. Eco-18-1237, Eco & Associates Inc. April 2019).

4. U.S. Environmental Protection Agency, Dec. 1996, *SW846 Laboratory Manual Physical/Chemical Methods.* Revision 3, Washington, D.C. 20460.

5. EM 200-1-10 Guidance for Evaluation Performance-based Chemical Data, US Army Corps of Engineers (USACE), June 2005.

6. Department of Defense Quality System Manual (DOD QSM), Version 5.3, 2019.

7. EPA Methods for Chemical Analysis of Water and Wastes. EPA -600-4-79-020. Revised; March 1983.