Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J009 Analytical Data Package

Publication Date: 02-23-2023

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 2022 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 10-03-22. EMAX Laboratories received the samples on 10-04-22. According to communication records, one sample (BGMW03102022, Lab ID#J009-04) was not analyzed due to being mistakenly preserved. Therefore, only eight samples were analyzed. 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 BGMW07102022 (Lab ID#J009-01) 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 analysis. Recoveries and results of LCS/LCSD was used to evaluate accuracy and precision. Raw data for method blanks and LCS/LCSDs were also 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 (8 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 # 22J009) 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 10-03-22. EMAX Laboratories received the samples on 10-04-22.

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 BGMW07102022 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 designated to be spiked as MS/MSD on the chain of custody. Raw data each set of LCS/LCSD was also 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#22J009 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
BGMW07102022	J009-01	10-03-22	S3VM	Anions by IC,
MW23102022	J009-02	10-03-22	S3VM	Anions by IC,
TMW18102022	J009-03	10-03-22	S3VM	Anions by IC,
BGMW03102022*	J009-04*	10-03-22	S3VM	Anions by IC,
BGMW03102022D	J009-05	10-03-22	S3VM	Anions by IC,
MW32102022	J009-06	10-03-22	S3VM	Anions by IC,
BGMW12102022	J009-07	10-03-22	S3VM	Anions by IC,
BGMW08102022	J009-08	10-03-22	S3VM	Anions by IC,
TMW36102022	J009-09	10-03-22	S3VM	Anions by IC,

*Sample was not analyzed;

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
		Analysis within 28 days for Bromide, Fluoride, Chloride and Sulfate	None. Holding times were met

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 3.8˚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:

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

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. The completeness objective is to obtain enough valid data to enable the goals and objectives of the project to be achieved. One sample (BGMW03102022) was not analyzed due to being mistakenly preserved. Only the corresponding field duplicate (BGMW03102022D) was analyzed.

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:

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

Completeness (EPA Method 9056A: Anions) =8/9X100=88.9%

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 eight 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 eight (8) water samples were collected on 10-03-22. Samples were analyzed on 10-04-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Some samples were re-analyzed on 10-05-22 and 10-20-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

4.12.2. 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 05-04-22 and 10-18-22. 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 05-04-22 and 10-18-22. Recoveries were all within

90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-04-22, 10-05-22 and 10-20-22. A total of ten 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 the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.12.3. Quality Control Samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. A second set of LCS/LCSD was analyzed with Chloride analysis. 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.

4.12.4. Field duplicate sample and its associated sample: Sample BGMW03102022 was identified as field duplicate of BGMW03102022. Due to field sample being mistakenly preserved, only field duplicate sample was analyzed. Therefore, no result was submitted for field sample (BGMW03102022).

4.12.5. Raw data was submitted for all requested field samples. Sample BGMW07102022 (Lab ID#22J009-01) 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 #22J009 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.

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J037 Analytical Data Package

Publication Date: 02-23-23

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 2022 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 fourteen (14) water samples were collected on 10-04-22. EMAX Laboratories received the samples on 10-05-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample MW02102022(Lab ID# J037-02) and TMW37102022(Lab ID# J037-04) were designated as stage 3 deliverable on the chain of custody. Raw data for these samples were compared to the reported summary tables for each method and went through comprehensive data validation review. No sample was designated to be spiked as MS/MSD on the chain of custody, therefore the results and recoveries of LCS/LCSD was used for both accuracy and precision. Raw data for method blank and LCS/LCSD for each method 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, (both extraction and analysis), extraction logs, 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 methods of analysis:

EPA Method 3050B/8260C: Volatile Organics by GC-MS (13 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (9 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (12 samples)
EPA Method SW8332: Nitroglycerine and PETN (12 samples)
EPA Method 8081B: Organochlorine Pesticides (10 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (4 sample)

EPA Method 8151A: Chlorinated herbicides (4 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (8 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (7 samples)
EPA Method 6850: Perchlorate (12 samples)
EPA Method 9056A: Anions by IC (13 samples)

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 Methods (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 extraction and analytical holding times were met for each method and all the related samples. The deviations, if any, are discussed in Section 4.0 for each method.

The analytical data evaluated in this data validation report (SDG # 22J037) has met the data quality and usability requirement as defined in the data quality objectives. While very few analytical QC exceedances were observed, it was not significant for any data qualifiers. 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 DoD *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 fourteen (14) water samples were collected on 10-04-22. EMAX Laboratories received the samples on 10-05-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the 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 preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), when applicable
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)
- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;

- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses 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. Next, selected analytical reports including QA/QC information was cross checked with raw data. The analysis and extraction sequence logs for each method were 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, surrogate recoveries, MS/MSD, and lab control samples (LCS/LCSD) 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 each analytical method requested on the chain of custody were submitted for all samples. Two field samples MW02102022 (EMAX ID #J037-02) and TMW37102022(Lab ID# J037-04) from this sample delivery group were designated as stage 3 data deliverable. Raw data for these samples were evaluated comprehensively. No sample was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank and LCS/LCSD was reviewed in detail. 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 regard to the requested method of analysis (collection to extraction and extraction 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
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated;
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards, and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

The following is a list of sample identifications and corresponding laboratory sample identification numbers:

Site Name: Fort Wingate, New Mexico

SDG#22J037 **Matrix: Water**

Field/Client ID	Lab ID	Date collected	Validation stage	Requested Methods of Analysis
MW28102022	22J037-01	10-04-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
MW02102022	22J037-02	10-04-22	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
TMW44102022	22J037-03	10-04-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Organochlorine Pesticides Perchlorate by 6850 Anions by IC
TMW37102022	22J037-04	10-04-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC
MW01102022	22J037-05	10-04-22	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22J037 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW46102022	22J037-06	10-04-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
TMW46102022D	22J037-07	10-04-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
TMW58102022	22J037-08	10-04-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
BGMW10102022	22J037-09	10-04-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22J037 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
BGMW03102022	22J037-10	10-04-22	S3VM	Anions by IC
SMW01102022	22J037-11	10-04-22		VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC
TMW26102022	22J037-12	10-04-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC
QC04102022EB1	22J037-13	10-04-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
QC04102022 TB2	22J037-14	10-04-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8330B	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in fifteen ice preserved coolers.

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction, and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements 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"
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	Not applicable
Anions by IC	Water	Analysis 48 hours from collection for Nitrate, Nitrite &Orthophosphate and 28-days for Bromide, Chloride, Fluoride and Sulfate	Holding times were met for all
Mercury & Dissolved Mercury	Water	Collection to Analysis: 28 days	Not applicable

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 and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in fifteen ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.4˚C and as high as 5.4˚C upon arrival. All samples were received intact and 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 exactly the same way. 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. The completeness objective is to obtain a sufficient amount 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:

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

Completeness (EPA Method 5030B/8260C: VOCs) =13/13X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =9/9X100=100%
Completeness (EPA Method 8330B: Explosives) =12/12X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =12/12X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =8/8X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =4/4X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =4/4X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 8/8X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =7/7X100=100%
Completeness (EPA Method 6850: Perchlorate) =12/12X100=100%
Completeness (EPA Method 9056A: Anions) =13/13X100=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 fourteen water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
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. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Thirteen water samples were collected on 10-04-22. Samples were analyzed on 10-10-22 and 10-11-22 within method's requirement for holding time. (Water samples were preserved with hydrochloric acid).

Chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.1.7.

4.1.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 10-06-22 and at the beginning of each analysis shift on 10-10-22 and 10-11-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 10-06-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table:

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 10-06-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.031) and 2-Butanone (0.015). However, recoveries were within the requirement of 70-130%. Calibration check compounds (CCCs) met the acceptance criteria for %RSD among the response factors calculated for each level. Table 4.1.3.2

lists the CCCs with method requirement limits for %RSD among response factors for initial calibration.

Table 4.1.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Response Factors %RSD 10-06-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 10-06-22
Chloromethane	0.999
p-Isopropyl toluene	0.9993
Ethyl methacrylate (non-target)	0.9990

4.1.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 10-06-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standard was analyzed at the beginning and end of each analysis shift on 10-10-22 and 10-11-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-10-22 (I &II)	Continuing cal. Response factors 10-11-22 (I&II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. The calculated % difference between RFs from continuing calibration and average response factors from initial calibration is summarized in Table 4.1.4.2 for continuing calibration reports presented with the data package.

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-10-22) I&II	%Deviation from Initial calibration (10-11-22) I&II
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from initial calibration was less than 20 percent for the rest of target list except for compounds listed in the table below for each daily standard:

Target Compounds	**%Deviation From Initial calibration (Acceptance Limit)**	**%Deviation from Initial calibration (10-10-22) %**		**%Deviation from Initial calibration (10-11-22) %**	
		I	II	I	II
2-Chloroethylvinyl ether	≤ 20%	42.3*	43.3*	49.6*	69.1*
1,2,3-Trichlorobenzene	≤ 20%	--	--	--	--
Dichlorodifluoromethane	≤ 20%	--	22.0*	--	--
Trichlorofluoromethane	≤ 20%	--	38.8*	33.0*	--
Acetone	≤ 20%	--	29.2*	--	36.0*
1,2,3-Trimethylbenzene	≤ 20%	--	21.6*	--	29.0*
1,1-Dichloroethane	≤ 20%	--	22.6*	--	20.5*
Carbon tetrachloride	≤ 20%	--	22.6*	--	--
Tetrahydrofuran	≤ 20%	--	--	22.6*	20.8*

*Outside control limits I: Opening Daily Std. II: Closing Daily Std.

Outliers are mostly in the closing daily standards. None of these compounds (except Acetone in equipment blank)) were detected in the corresponding field samples. This should not affect the data quality.

4.1.5. Quality Control samples reported consisted of two method blanks and two sets of LCS/LCSD only. No sample was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.
Surrogate recoveries were all within the method's acceptable limits.

4.1.6. **Field duplicate sample** and its associated sample: Field sample TMW46102022 was identified as field duplicate of sample TMW46102022D. No Volatile Organic Compounds was detected in the sample and associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Samples MW02102022 (Lab ID# 22J037-02) and TMW37102022 (Lab ID# 22J037-04) were designated to be reviewed as stage 3 data deliverable. Raw data for these samples with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

4.2. SVOC (EPA Method 3520C/8270D)

4.2.1. Technical Holding Times

Holding time requirement was met for all nine samples. Water samples were collected on 10-04-22, extracted on 10-08-22 and were analyzed on 10-14-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 10-14-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group.

Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table.

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-07-22
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Average response factors for the rest of target compounds were within method's recommended values.

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 20%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-07-22
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 03-07-22
Benzoic acid	0.9969
2,4-Dinitrophenol	0.9964
4-Nitrophenol	0.9971
Di-n-Octyl phthalate	0.9972

All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-14-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (10-14-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for all other target

analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Percent difference between RFs from continuing calibration and average response factors from initial calibration passed the method's criteria as summarized in Table 4.2.4.2.

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (10-14-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds in closing daily standard as listed in the table below:

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-14-22) %	
		I	II
2,4-Dinitrophenol	≤ 20%	29.0*	--
2-Methylphenol	≤ 20%	--	29.1*
Bis(2-Chloroisopropyl) ether	≤ 20%	--	28.4*
4-Methylphenol	≤ 20%	--	24.4*
4-Nitroaniline	≤ 20%	--	29.2*
Pentachlorophenol	≤ 20%	--	25.5*
Bis (2-Ethylhexylphthalate)	≤ 20%	--	27.6*
Caprolactam	≤ 20%	--	25.6*

4.2.5. Quality Control samples reported consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were mostly within the project acceptance limits, except for one compound as listed in the table below:

Compound	LCS%	LCSD%	**QC Limit%**
Bis (2-Ethylhexylphthalate)	140*	140*	55-135

*Outside QC limit

This compound was not detected in any of the field samples; therefore, it should not affect the data quality.

Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision for the rest of target list in this method. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method's acceptable limits.

4.2.6. **Field duplicate sample** and its associated sample: Field sample TMW46102022 was identified as field duplicate of sample TMW46102022D. No Semi Volatile Organic Compounds was detected in the sample and associated field duplicate sample.

4.2.7. Raw data was submitted for all samples. Sample TMW37042022(Lab ID#22J037-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.3. ORGANOCHLORINE PESTICIDES (EPA Method 3520C/ 8081B)

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all ten water samples requested for this method. Water samples were collected on

10-04-22, extracted on 10-08-22 and analyzed on 10-12-22 within required holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 09-02-22, 09-12-22 and before sample analysis on 10-12-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 09-02-22 and 09-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 08-11-22 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene.

Retention time window width were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 08-11-22 and 09-12-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Performance of instrument was monitored by analysis of DDT and Endrin breakdown mixture. Before each continuing (daily) calibration a mixture of DDT and Endrin was analyzed.

Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval on 10-12-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. In the three continuing calibration standards, one mid-point concentration of 20-40μg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD only. No sample was designated to be spiked for MS/MSD for this method. The full pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits. Results and recoveries of QC samples were confirmed with the reported raw data. Results for method blank were reviewed for each component and no organochlorine pesticide was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Field sample TMW46102022 was identified as field duplicate of sample TMW46102022D. No Pesticides was detected in the sample and associated field duplicate sample.

4.3.6. Raw data was submitted for all samples. Samples MW02102022(Lab ID#22J037-02) and TMW37102022(Lab ID#22J037-04) were designated as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. Polychlorinated Biphenyls (EPA Method 3520C/ 8082A)

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Four water samples were collected on 10-04-22, extracted on 10-08-22, and analyzed on 10-13-22, within holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 09-07-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

Retention time window width were established at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 09-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-13-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD, therefore results and recoveries of LCS/LCSD was used for both accuracy and precision. Percent recoveries (%R) were within the established QC limits. However, percent RPD for Aroclor-1016 was not within QC limit (45%*).

Results for method blank was reviewed for each component and no target analyte was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample: No field duplicate sample was assigned to this method.

4.4.6. Raw data was submitted for all samples. Sample MW28102022(Lab ID#22J037-01) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Four water samples were collected on 10-04-22, extracted on 10-07-22 and analyzed on 10-13-22.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 10-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among

calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99819 was used for MCPP in column B.

Retention time windows were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 10-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-13-22 and 10-14-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels.

Results for surrogate recoveries and QC were all reported from channel A. Channel B was used for confirmation only.

4.5.4. Quality Control samples consisted of one method blank, and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. All herbicides target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits

Results for method blank was reviewed for each component and no Herbicide was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.5.5. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this method.

4.5.6. Raw data was submitted for all samples. Sample MW28102022(Lab ID#22J037-01) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Nitroaromatics by HPLC/UV (EPA Method 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 10-04-22, extracted on 10-08-22 and analyzed on 10-10-22 and 10-11-22, within holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis. Positive results, if any, were confirmed with UHPLC equipped with different column (Kinetex- Biphenyl column).

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-25-22 and 08-26-22. Confirmation (Kinetex-Biphenyl column) was calibrated on 08-23-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

Retention time windows were established for each target analyte at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for both columns on 08-23-21 and 08-26-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of four continuing calibration standards were analyzed on 10-10-22 and 10-11-22, bracketing the analyses of samples and all the QC samples. Three more daily standards were analyzed with the confirmation column on 10-17-22. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.6.5 Field duplicate sample and its associated sample**:** Sample TMW46102022 was identified as field duplicate of sample TMW46102022D. No explosive target compound was detected in each sample and field duplicate sample.

4.6.6. Raw data was submitted for all samples. Samples MW02102022(Lab ID#J037-02) and TMW37102022(Lab ID#J037-04) were designated to be reviewed as stage 3 data deliverable. Raw data for these samples together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation (Biphenyl column).

4.7. Nitroglycerine and PETN by UHPLC/UV (EPA Method 8332)

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 10-04-22, extracted on 10-08-22 and analyzed on 10-10-22.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis.

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 08-24-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time windows were established for each target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 08-24-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-10-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Sample TMW46102022 was identified as field duplicate of sample TMW46102022D. No explosive target compound was detected in sample and field duplicate sample.

4.7.6. Raw data was submitted for all samples. Samples MW02102022(Lab ID#J037-02) and TMW37102022(Lab ID#J037-04) were designated to be reviewed as stage 3 data deliverable. Raw data for these samples together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.8. Total Petroleum hydrocarbons GRO (EPA Method 8015G)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight water samples requested for this method. Water samples were collected on 10-04-22. Samples were analyzed on 10-12-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Sample was carried through the system by purge and trap.

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-06-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10). Retention times for further sample analyses was used for peak identification and integration range.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-06-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-12-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank.
Surrogate recoveries were all within the method QC acceptance limits.

4.8.5. Field duplicate sample and its associated sample: Field sample TMW46102022 was identified as field duplicate of sample TMW46102022D. No TPH as Gasoline was detected in the sample and associated field duplicate sample.

4.8.6. Raw data was submitted for all samples. Sample MW02102022(Lab ID#22J037-02) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven water sample requested for this method. Water samples were collected on 10-04-22, extracted on 10-07-22 and analyzed on 10-10-22.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Heavier range of total petroleum hydrocarbons were extracted and introduced into system by direct injection.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-08-22. Calibration factor (area for each compound/concentration) was used to quantify diesel range hydrocarbons (TPH as DRO). A second set of initial calibration curve was generated for lighter TPHs (Jet Fuel) and heavier TPHs (motor oil). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time window width was established by analysis of a window defining hydrocarbon standard (C10-C40). Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-08-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-10-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated for MS/MSD. Percent recoveries (%R) of LCS/LCSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.9.5. Field duplicate sample and its associated sample: Field sample MW46102022 was identified as field duplicate of sample TMW46102022D. No TPH as Diesel was detected in the sample and associated field duplicate sample.

4.9.6. Raw data was submitted for all samples. Sample MW02102022(Lab ID#22J037-02) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.10. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Twelve (12) water samples were collected on 10-04-22. Samples were analyzed on 10-05-22 and 10-06-22.

A High-Performance LC coupled with Mass Detector (HPLC/MS) was used for analysis.

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-21-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9960 (Perchlorate ion 83) and 0.9987 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

Retention time for each isotope (ion 83 and 85) at each calibration level was within 0.2 minutes required by the method.

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-21-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of nine daily standards were carried out on 10-05-22 and 10-06-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW58102022 was selected to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets of LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. No surrogate is used in this method.

4.10.5. Field duplicate sample and its associated sample: Sample TMW46102022 was identified as field duplicate of TMW46102022D. No perchlorate was detected in each sample and associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Samples MW02102022(Lab ID#J037-02) and TMW37102022(Lab ID#J037-04) were designated to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of water samples requested for this method. A total of thirteen (13) water samples were collected on 10-04-22. Samples were analyzed on 10-05-22 and 10-06-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Samples were re-analyzed with dilution on 10-21-22 and 10-22-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

4.11.2. 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 05-04-22 and 10-18-22. 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 05-04-22 and 10-18-22. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-05-22, 10-06-22, 10-21-22and 10-22-22. A total of eleven 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.

4.11.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD, MS/MSD and sample/sample duplicate. Sample TMW58102022 was selected to be spiked as MS/MSD and sample/sample duplicate. One more set of MB and LCS/LCSD was analyzed with Chloride and Sulfate re- analysis on 10-21-22. 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.

Recoveries of MS/MSD were mostly within acceptable limits except for few anions as indicated in the table below:

Anion	TMW58102022 (Lab ID#J037-08) MS%	TMW58102022 (Lab ID#J037-08) MSD%	% Acceptance limits
Bromide	87*	87*	91-110
Nitrate	85*	84*	88-111
Nitrite	131*	132*	87-111
O-Phosphate	63*	63*	80-116
Sulfate	137*	151*	87-112

*Outside Acceptance limits

4.11.4. Field duplicate sample and its associated sample: Sample TMW46102022 was identified as field duplicate of TMW46102022D. Results for sample/sample duplicate is summarized in the table below:

Anion	TMW46102022 (Lab ID #J037-06) mg/L	TMW46102022D (Lab ID #J037-07) mg/L	**% RPD**
Nitrate	70	74	5.56
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	1.3	1.3	<1
Chloride	230	260	12.2
Fluoride	0.30	0.30	<1
Sulfate	1700	1800	5.71

4.11.5. Raw data was submitted for all requested field samples. Samples MW02102022 (Lab ID#22J037-02) and TMW37102022 (Lab ID#22J037-04) were designated to be reviewed as stage 3 data deliverable. Raw data for these samples 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 #22J037 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

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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J039 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 2022 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 twelve (12) water samples were collected on 10-03-22. EMAX Laboratories received the samples on 10-05-22. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample MW32102022(Lab ID# J039-07) was selected to be reviewed as stage 3 deliverable. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation review. No sample was designated to be spiked as MS/MSD on the chain of custody, therefore the results and recoveries of LCS/LCSD was used for both accuracy and precision. Raw data for method blank and LCS/LCSD for each method 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, (both extraction and analysis), extraction logs, 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 methods of analysis:

EPA Method 3050B/8260C: Volatile Organics by GC-MS (12 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (11 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (11 samples)
EPA Method SW8332: Nitroglycerine and PETN (11 samples)
EPA Method 8081B: Organochlorine Pesticides (8 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (4 sample)
EPA Method 8151A: Chlorinated herbicides (4 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (3 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (2 samples)
EPA Method 6850: Perchlorate (11 samples)

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 Methods (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 extraction and analytical holding times were met for each method and all the related samples. The deviations, if any, are discussed in Section 4.0 for each method.

The analytical data evaluated in this data validation report (SDG # 22J039) has met the data quality and usability requirement as defined in the data quality objectives. While very few analytical QC exceedances were observed, it was not significant for any data qualifiers. 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 DoD *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. Method SW846 3535A/8330B

A total of twelve (12) water samples were collected on 10-03-22. EMAX Laboratories received the samples on 10-05-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the 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 preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if applicable
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)
- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;

- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses 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. Next, selected analytical reports including QA/QC information was cross checked with raw data. The analysis and extraction sequence logs for each method were 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, surrogate recoveries, MS/MSD, and lab control samples (LCS/LCSD) 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 each analytical method requested on the chain of custody were submitted for all samples. One field sample MW32102022 (EMAX ID #J039-07) from this sample delivery group was reviewed as stage 3 data deliverable. Raw data for this sample was evaluated comprehensively. No sample was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank and LCS/LCSD was reviewed in detail. 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 regard to the requested method of analysis (collection to extraction and extraction 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
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated;
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards, and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

The following is a list of sample identifications and corresponding laboratory sample identification numbers:

Site Name: Fort Wingate, New Mexico

SDG#22J039 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
BGMW07102022	22J039-01	10-03-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850
TMW16102022	22J039-02	10-03-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
MW23102022	22J039-03	10-03-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Organochlorine Pesticides Perchlorate by 6850
TMW18102022	22J039-04	10-03-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
BGMW03102022	22J039-05	10-03-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#22J039 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
BGMW03102022D	22J039-06	10-03-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
MW32102022	22J039-07	10-03-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850
TMW19102022	22J039-08	10-03-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
BGMW12102022	22J039-09	10-03-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#22J037 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
BGMW08102022	22J039-10	10-03-22		VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850
TMW36102022	22J039-11	10-03-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
QC10032022 TB1	22J039-12	10-03-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 μg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20μg/L, (Benzidine=40μg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4μg/L
	Nitroglycerine & PETN	SW8330B	120μg/L
	Chlorinated Herbicides	SW8151A	1μg/L, (MCPA=40μg/L)
	Organochlorine Pesticides	SW8081B	0.1μg/L Methoxychlor =1.0μg/L Toxaphene =2.0μg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1μg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100μg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5μg/L,1μg/L,20μg/L,100μg/L,200μg/L
	Dissolved Mercury/Mercury	SW7470A	0.5μg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2μg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in twelve ice preserved coolers.

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction, and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements 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"
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	Not applicable
Anions by IC	Water	Analysis 48 hours from collection for Nitrate, Nitrite &Orthophosphate and 28-days for Bromide, Chloride, Fluoride and Sulfate	Not applicable
Mercury & Dissolved Mercury	Water	Collection to Analysis: 28 days	Not applicable

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 and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in twelve ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.4˚C and as high as 3.4˚C upon arrival. All samples were received intact and 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 exactly the same way. 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:

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

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. The completeness objective is to obtain a sufficient amount 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 5030B/8260C: VOCs) =12/12X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =11/11X100=100%

Completeness (EPA Method 8330B: Explosives) =11/11X100=100%

Completeness (EPA Method 8332: Nitroglycerine & PETN) =11/11X100=100%

Completeness (EPA Method 8081B: Organochlorine pesticides) =8/8X100=100%

Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =4/4X100=100%

Completeness (EPA Method 8151B: Chlorinated Herbicides) =4/4X100=100%

Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 3/3X100=100%

Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =2/2X100=100%
Completeness (EPA Method 6850: Perchlorate) =11/11X100=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 twelve water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS

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. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Twelve water samples were collected on 10-03-22. Samples were analyzed on 10-10-22 and 10-11-22 within method's requirement for holding time. (Water samples were preserved with hydrochloric acid).

Chain-of-custody was reviewed for documentation of sample information and method of analysis. Qualification notations, if any, will be summarized in result section; section 4.1.7.

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 08-24-22 and at the beginning of analysis shift on 10-10-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 08-24-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table:

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 08-24-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.056) and 2-Butanone (0.056). However, recoveries were within the requirement of 70-130%. Calibration check compounds (CCCs) met the acceptance criteria for %RSD among the response factors calculated for each level. Table 4.1.3.2 lists the CCCs with method requirement limits for %RSD among response factors for initial calibration.

Table 4.1.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Response Factors %RSD 08-24-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 08-24-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standard was analyzed at the beginning and end of analysis shift on 10-10-22 and 10-11-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-10-22 (Opening)	Continuing cal. Response factors 10-11-22 (Closing)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for

all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Calculated % difference between RFs from continuing calibration and average response factors from initial calibration is summarized in Table 4.1.4.2 for continuing calibration reports presented with the data package.

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration 10-10-22 (Opening)	%Deviation from Initial calibration 10-11-22 (Closing)
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

4.1.5. Quality Control samples reported consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.
Surrogate recoveries were all within the method's acceptable limits.

4.1.6. **Field duplicate sample** and its associated sample: Field sample BGMW03102022 was identified as field duplicate of sample BGMW03102022D. No Volatile Organic Compounds was detected in the sample and associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample MW32102022 (Lab ID# 22J039-07) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with all related

QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

4.2. SVOC (EPA Method 3520C/8270D)

4.2.1. Technical Holding Times

Holding time requirement was met for all eleven samples. Water samples were collected on 10-03-22, extracted on 10-08-22 and were analyzed on 10-14-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 10-14-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group.

Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table.

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-07-22
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Average response factors for the rest of target compounds were within method's recommended values.

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 20%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-07-22
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 03-07-22
Benzoic acid	0.9969
2,4-Dinitrophenol	0.9964
4-Nitrophenol	0.9971
Di-n-Octyl phthalate	0.9972

All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-14-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (10-14-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Percent difference between RFs from continuing calibration and average response factors from initial calibration passed the method's criteria as summarized in Table 4.2.4.2.

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (10-14-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds from closing daily standard as listed in the table below:

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-14-22) %	
		I	II
2,4-Dinitrophenol	≤ 20%	29.0*	--
2-Methylphenol	≤ 20%	--	29.1*
Bis(2-Chloroisopropyl) ether	≤ 20%	--	28.4*
4-Methylphenol	≤ 20%	--	24.4*
4-Nitroaniline	≤ 20%	--	29.2*
Pentachlorophenol	≤ 20%	--	25.5*
Bis (2-Ethylhexylphthalate)	≤ 20%	--	27.6*
Caprolactam	≤ 20%	--	25.6*

4.2.5. Quality Control samples reported consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits, except for one compound as listed in the table below:

Compound	LCS%	LCSD%	**QC Limit%**
Bis (2-Ethylhexylphthalate)	140*	140*	55-135

*Outside QC limit

This compound was not detected in any of the field samples; therefore, it should not affect the data quality.

Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision for the rest of target list in this method. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method’s acceptable limits.

4.2.6. **Field duplicate sample** and its associated sample: Field sample BGMW03102022 was identified as field duplicate of sample BGMW03102022D. No Semi Volatile Organic Compounds was detected in the sample and associated field duplicate sample.

4.2.7. Raw data was submitted for all samples. Sample MW32042022(Lab ID#22J039-07) was reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.3. ORGANOCHLORINE PESTICIDES (EPA Method 3520C/ 8081B)

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight water samples requested for this method. Water samples were collected on

10-03-22, extracted on 10-07-22 and analyzed on 10-11-22 within required holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 09-02-22, 09-12-22 and before sample analysis on 10-11-22,10-12-22 and 10-13-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 09-02-22 and 09-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 08-11-22 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene.

Retention time window width were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 08-11-22 and 09-12-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Performance of instrument was monitored by analysis of DDT and Endrin breakdown mixture. Before each continuing (daily) calibration a mixture of DDT and Endrin was analyzed.

Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of four continuing calibration standards were analyzed at 10-injections interval on 10-11-22,10-12-22 and 10-13-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. In four continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD only. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits. Results and recoveries of QC samples were confirmed with the reported raw data.

Results for method blank were reviewed for each component and no organochlorine pesticide was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Field sample BGMW03102022 was identified as field duplicate of sample BGMW03102022D. No Pesticide was detected in the sample and associated field duplicate sample.

4.3.6. Raw data was submitted for all samples. Sample MW32102022(Lab ID#22J039-07) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. Polychlorinated Biphenyls (EPA Method 3520C/ 8082A)

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Four water samples were collected on 10-03-22, extracted on 10-07-22, and analyzed on 10-12-22, within holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 09-07-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

Retention time window width were established at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 09-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-12-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD, therefore results and recoveries of LCS/LCSD was used for both accuracy and precision. Percent recoveries (%R) were within the established QC limits.

Results for method blank was reviewed for each component and no target analyte was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample: No field duplicate sample was assigned to this method.

4.4.6. Raw data was submitted for all samples. Sample MW32102022(Lab ID#22J039-07) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Four water samples were collected on 10-03-22, extracted on 10-07-22 and analyzed on 10-13-22 and 10-14-22.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 10-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among

calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99819 was used for MCPP in column B.

Retention time windows were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 10-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-13-22 and 10-14-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels.

Results for surrogate recoveries and QC were all reported from channel A. Channel B was used for confirmation only.

4.5.4. Quality Control samples consisted of one method blank, and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. All herbicides target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits

Results for method blank was reviewed for each component and no Herbicide was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.5.5. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this method.

4.5.6. Raw data was submitted for all samples. Sample MW32102022(Lab ID#22J039-07) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Nitroaromatics by HPLC/UV (EPA Method 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eleven (11) water samples requested for this method. Water samples were collected on 10-03-22, extracted on 10-07-22 and analyzed on 10-10-22 and 10-11-22, within holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis. Positive results, if any, were confirmed with UHPLC equipped with different column (Kinetex- Biphenyl column).

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-25-22 and 08-26-22. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

Retention time windows were established for each target analyte at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for on 08-26-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of five continuing calibration standards were analyzed on 10-10-22 and 10-11-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.6.5 Field duplicate sample and its associated sample**:** Sample BGMW03102022 was identified as field duplicate of sample BGMW03102022D. No explosive target compound was detected in each sample and field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample MW32102022(Lab ID#J039-07) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation (Biphenyl column).

4.7. Nitroglycerine and PETN by UHPLC/UV (EPA Method 8332)

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eleven (11) water samples requested for this method. Water samples were collected on 10-03-22, extracted on 10-07-22 and analyzed on 10-10-22.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis.

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on

08-24-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time windows were established for each target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 08-24-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of five continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-10-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Sample BGMW03102022 was identified as field duplicate of sample BGMW03102022D. No explosive target compound was detected in sample and field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample MW32102022(Lab ID#J039-07) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.8. Total Petroleum hydrocarbons GRO (EPA Method 8015G)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for three water samples requested for this method. Water samples were collected on 10-03-22. Samples were analyzed on 10-11-22 and 10-12-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Sample was carried through the system by purge and trap.

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-06-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10). Retention times for further sample analyses was used for peak identification and integration range.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-06-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-11-22 and 10-12-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank.
Surrogate recoveries were all within the method QC acceptance limits.

4.8.5. Field duplicate sample and its associated sample: No field duplicate sample was assigned for this method,

4.8.6. Raw data was submitted for all samples. Sample MW32102022(Lab ID#22J039-07) was reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for two water sample requested for this method. Water samples were collected on 10-03-22, extracted on 10-07-22 and analyzed on 10-10-22.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Heavier range of total petroleum hydrocarbons were extracted and introduced into system by direct injection.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-08-22. Calibration factor (area for each compound/concentration) was used to quantify diesel range hydrocarbons (TPH as DRO). A second set of initial calibration curve was generated for lighter TPHs (Jet Fuel) and heavier TPHs (motor oil). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time window width was established by analysis of a window defining hydrocarbon standard (C10-C40). Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-08-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-10-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated for MS/MSD. Percent recoveries (%R) of LCS/LCSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.9.5. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this method.

4.9.6. Raw data was submitted for all samples. Sample MW32102022(Lab ID#22J039-07) was reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.10. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Eleven (11) water samples were collected on 10-03-22. Samples were analyzed on 10-05-22 and 10-06-22.

A High-Performance LC coupled with Mass Detector (HPLC/MS) was used for analysis.

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-21-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9960 (Perchlorate ion 83) and 0.9987 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

Retention time for each isotope (ion 83 and 85) at each calibration level was within 0.2 minutes required by the method.

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-21-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of twelve daily standards were carried out on 10-05-22 and 10-06-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of two method blanks and two sets of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established QC limits for both sets of LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. No surrogate is used in this method.

4.10.5. Field duplicate sample and its associated sample: Sample BGMW03102022 was identified as field duplicate of BGMW03102022D. No perchlorate was detected in each sample and associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample MW32102022(Lab ID#J039-07) was selected to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #22J039 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

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Final Data Validation Report

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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 2022 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 thirteen (13) water samples were collected on 10-04-22 and 10-05-22. EMAX Laboratories received the samples on 10-06-22. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample TMW07102022(Lab ID# J051-02) was designated as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation review. Samples TMW63102022 and TMW27102022 were designated to be spiked as MS/MSD on the chain of custody. Results and recoveries of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision. Raw data for method blank and LCS/LCSD and MS/MSD for each method 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, (both extraction and analysis), extraction logs, 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 methods of analysis:

EPA Method 3050B/8260C: Volatile Organics by GC-MS (13 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (10 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (10 samples)
EPA Method SW8332: Nitroglycerine and PETN (10 samples)
EPA Method 8081B: Organochlorine Pesticides (10 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (6 sample)

EPA Method 8151A: Chlorinated herbicides (6 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (9 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (8 samples)
EPA Method 6850: Perchlorate (11 samples)
EPA Method 9056A: Anions by IC (11 samples)

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 Methods (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 extraction and analytical holding times were met for each method and all the related samples. The deviations, if any, are discussed in Section 4.0 for each method.

The analytical data evaluated in this data validation report (SDG # 22J051) has met the data quality and usability requirement as defined in the data quality objectives. While very few analytical QC exceedances were observed, it was not significant for any data qualifiers. 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 DoD *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 thirteen (13) water samples were collected on 10-04-22 and 10-05-22. EMAX Laboratories received the samples on 10-06-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the 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 preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD)
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses 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. Next, selected analytical reports including QA/QC information was cross checked with raw data. The analysis and extraction sequence logs for each method were 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, surrogate recoveries, MS/MSD, and lab control samples (LCS/LCSD) 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 each analytical method requested on the chain of custody were submitted for all samples. One field sample TMW07102022(Lab ID# J051-02) from this sample delivery group was designated as stage 3 data deliverable on the chain of custody. Raw data for this sample was evaluated comprehensively. Samples TMW63102022 and TMW27102022 were designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank and LCS/LCSD and MS/MSD was reviewed in detail. 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 regard to the requested method of analysis (collection to extraction and extraction 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
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated;
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards, and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

The following is a list of sample identifications and corresponding laboratory sample identification numbers:

Site Name: Fort Wingate, New Mexico

SDG#22J051 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW29102022	22J051-01	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
TMW07102022	22J051-02	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN TPH Gasoline; TPH as DRO Anions by IC
BGMW01102022	22J051-03	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC
TMW30102022	22J051-04	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC
BGMW02102022	22J051-05	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22J051 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW50102022	22J051-06	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
TMW08102022	22J051-07	10-05-22	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC
TMW63102022	22J051-08	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
QC05102022EB2	22J051-09	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
TMW51102022	22J051-10	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22J051 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW51102022D	22J051-11	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
TMW27102022	22J051-12	10-04-22	S3VM	VOCs by SW5030B/8260C, Perchlorate by 6850
QC05102022 TB3	22J051-13	10-05-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW63102022MS	22J051-08M	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
TMW63102022MSD	22J051-08S	10-05-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC
TMW27102022MS	22J051-12M	10-04-22	S3VM	VOCs by SW5030B/8260C, Perchlorate by 6850
TMW27102022MSD	22J051-12S	10-04-22	S3VM	VOCs by SW5030B/8260C, Perchlorate by 6850

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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8330B	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in eighteen ice preserved coolers.

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction, and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements 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"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	NA
Anions by IC	Water	Analysis 48 hours from collection for Nitrate, Nitrite &Orthophosphate and 28-days for Bromide, Chloride, Fluoride and Sulfate	Holding times were met for all
Mercury & Dissolved Mercury	Water	Collection to Analysis: 28 days	NA

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 and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in eighteen ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 0.3˚C and as high as 3.3˚C upon arrival. All samples were received intact and 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 exactly the same way. 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:

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

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. The completeness objective is to obtain a sufficient amount 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 5030B/8260C: VOCs) =13/13X100=100%

Completeness (EPA Method 3520B/8270D: SVOCs) =10/10X100=100%

Completeness (EPA Method 8330B: Explosives) =10/10X100=100%

Completeness (EPA Method 8332: Nitroglycerine & PETN) =10/10X100=100%

Completeness (EPA Method 8081B: Organochlorine pesticides) =10/10X100=100%

Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =6/6X100=100%

Completeness (EPA Method 8151B: Chlorinated Herbicides) =6/6X100=100%

Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 9/9X100=100%

Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =8/8X100=100%

Completeness (EPA Method 6850: Perchlorate) =11/11X100=100%

Completeness (EPA Method 9056A: Anions) =11/11X100=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 thirteen water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
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. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Thirteen water samples were collected on 10-04-22 and 10-05-22. Samples were analyzed on 10-13-22, 10-18-22 and 10-19-22 within method's requirement for holding time. (Water samples were preserved with hydrochloric acid).

Chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.1.7.

4.1.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to each initial calibration on 09-22-22, 10-14-22, 10-15-22 and at the beginning of each analysis shift on 10-13-22, 10-18-22 and 10-19-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to three sets of initial calibration using GC/MSD. Initial calibration curves were generated on 09-22-22, 10-14-22 and 10-15-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table:

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 09-22-22	Average Response factor 10-14-22	Average Response factor 10-15-22
Chloromethane	≥ 0.10	√	√	√
1,1-Dichloroethane	≥ 0.20	√	√	√
Bromoform	≥ 0.10	√	√	√
Chlorobenzene	≥ 0.50	√	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone and 2-Butanone as summarized n the table below:

Target compounds	Average Response factor 09-22-22	Average Response factor 10-14-22	Average Response factor 10-15-22
Acetone	0.043*	0.027*	0.045*
2-Butanone	0.017*	0.009*	0.087*

*Failed minimum average response factor of 0.10

However, recoveries were within the requirement of 70-130%. Calibration check compounds (CCCs) met the acceptance criteria for %RSD among the response factors calculated for each level. Table 4.1.3.2 lists the CCCs with method requirement limits for %RSD among response factors for initial calibration.

Table 4.1.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Response Factors %RSD 09-22-22	Response Factors %RSD 10-14-22	Response Factors %RSD 10-15-22
Vinyl chloride	-≤ 20%	√	√	√
1,1-Dichloroethene	-≤ 20%	√	√	√
Chloroform	-≤ 20%	√	√	√
1,2-Dichloropropane	-≤ 20%	√	√	√
Toluene	-≤ 20%	√	√	√
Ethyl benzene	-≤ 20%	√	√	√

√ denotes passing method acceptance limits

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 09-24-22	Least Square Linear Regression (CCF) 10-14-22
2-Chloroethylvinyl ether	0.9984	√
1-Chloroethane	0.9986	√
Acetone	√	0.9992

4.1.4. Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard on 09-22-22, 10-14-22 and 10-15-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standard was analyzed at the beginning and end of each analysis shift on 10-13-22, 10-18-22 and 10-19-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-13-22 (I &II)	Continuing cal. Response factors 10-18-22 (I&II)	Continuing cal. Response factors 10-19-22 (I&II)
Chloromethane	≥ 0.10	√	√	√
1,1-Dichloroethane	≥ 0.20	√	√	√
Chlorobenzene	≥ 0.50	√	√	√
Bromoform	≥ 0.10	√	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. The calculated % difference between RFs from continuing calibration and average response factors from initial calibration is summarized in Table 4.1.4.2 for continuing calibration reports presented with the data package.

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-13-22) I&II	%Deviation from Initial calibration (10-18-22) I&II	%Deviation from Initial calibration (10-19-22) I&II
Vinyl chloride	≤ 20%	√	√	√
1,1-Dichloroethene	≤ 20%	√	√	√
Chloroform	≤ 20%	√	√	√
		√	√	√

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-13-22) I&II	%Deviation from Initial calibration (10-18-22) I&II	%Deviation from Initial calibration (10-19-22) I&II
1,2-Dichloropropane	≤ 20%	√	√	√
Toluene	≤ 20%	√	√	√
Ethyl benzene	≤ 20%			

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list except for compounds in the table below for each daily standard:

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-13-22) %		%Deviation from Initial calibration (10-18-22) %		%Deviation from Initial calibration (10-19-22) %	
		opening	Closing	opening	closing	opening	closing
2-Chlorotoluene	≤ 20%	23.5*	--	√	√	√	√
Sec. Butylbenzene	≤ 20%	√--	21.3*	41.9*	√	√	√
2-Chloroethylvinyl ether	≤ 20%	√--	--	√	√	√	√
Bromomethane	≤ 20%	√--	--	√	√	√	26.7*
Chloroethane	≤ 20%	√--	--	√	√	√	22.9*
Dichlorofluoromethane	≤ 20%	√	--	√	√	√	25.1*
Trichlorofluoromethane	≤ 20%	√	--	√	√	√	23.6*
1,2-Dichloroethane	≤ 20%	√	--	√	√	√	20.9*
1,2,4-Trichlorobenzene	≤ 20%	√	--	√	√	√	22.1*
1,2,3-Trichlorobenzene	≤ 20%	√	--	√	√	√	24.5*

*Outside control limits

The outliers are mostly in the closing daily standards. None of these compounds were detected in the corresponding field samples. This should not affect the data quality.

4.1.5. Quality Control samples reported consisted of three method blanks, three sets of LCS/LCSD and two sets of MS/MSD. Samples TMW63102022 (Lab ID# J051-08) and TMW27102022 (Lab ID# J051-12) were designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list, except for the compounds listed in the table below:

VOC Target Compound	TMW63102022 (J051-08) MS%	TMW63102022 (J051-08) MSD%	**%** Acceptance limits
2-Chlorotoluene	117	123*	79-122
Sec.Butylbenze	122	130*	77-126

*Outside control limits

These compounds were not detected in the corresponding field samples, therefore, would not affect the data quality.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blanks presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method's acceptable limits.

4.1.6. **Field duplicate sample** and its associated sample: Field sample TMW51102022 was identified as field duplicate of sample TMW51102022D. No Volatile Organic Compounds (VOCs) was detected in the sample and associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample TMW07102022 (Lab ID# 22J051-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

4.2. SVOC (EPA Method 3520C/8270D)

4.2.1. Technical Holding Times

Holding time requirement was met for all nine samples. Water samples were collected on 10-05-22, extracted on 10-11-22 and were analyzed on 10-17-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 10-17-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group.

Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table.

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-07-22
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Average response factors for the rest of target compounds were within method's recommended values.

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 20%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-07-22
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 03-07-22
Benzoic acid	0.9969
2,4-Dinitrophenol	0.9964
4-Nitrophenol	0.9971
Di-n-Octyl phthalate	0.9972

All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-17-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (10-17-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Percent difference between RFs from continuing calibration and average response factors from initial calibration passed the method's criteria as summarized in Table 4.2.4.2.

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (10-17-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√

Calibration Check Compounds (CCCs)	**% Deviation From Initial calibration (Acceptance Limit)**	**Accepted Deviation from Initial calibration (10-17-22) I& II**
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds from closing daily standard as listed in the table below:

Target Compounds	**%Deviation From Initial calibration (Acceptance Limit)**	**%Deviation from Initial calibration (10-17-22) %**	
		I	II
2,4-Dinitrophenol	≤ 20%	30.5*	√
1,4-Dinitrobenzene	≤ 20%	√	23.8*
1,3-Dinitrobenzene	≤ 20%	√	21.1*

*Outside control limits

4.2.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63102022 was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for all the QC samples reported were mostly within the project acceptance limits, except for compounds listed in the table below:

Compound	TMW63102022J (051-08) MS%	TMW63102022 (J051-08) MSD%	**QC Limit%**
Bis (2-Ethylhexylphthalate)	136*	125	55-135
4-Nitroaniline	135*	109	60-130
Chrysene	124*	116	59-123

*Outside QC limit

RPD limits exceeded maximum 20% in Hexachlorobutadiene (24%), Hexachloroethane (21%) and 4-Nitroaniline (31%).

These compounds were not detected in any of the field samples; therefore, it should not affect the data quality.

Results and recoveries of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision for the rest of target list in this method.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method's acceptable limits.

4.2.6. **Field duplicate sample** and its associated sample: Field sample TMW51102022 was identified as field duplicate of sample TMW51102022D. No Semi Volatile Organic Compounds was detected in the sample and associated field duplicate sample.

4.2.7. Raw data was submitted for all samples. Sample TMW07042022(Lab ID#22J051-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.3. ORGANOCHLORINE PESTICIDES (EPA Method 3520C/ 8081B)

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all ten water samples requested for this method. Water samples were collected on 10-05-22, extracted on 10-10-22 and analyzed on 10-17-22 within required holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 03-11-22, 03-12-22 and before sample analysis on 10-16-22 and 10-17-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 03-11-22 and 03-12-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 03-12-22 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene.

Retention time window width were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 03-12-22 and 03-15-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Performance of instrument was monitored by analysis of DDT and Endrin breakdown mixture. Before each continuing (daily) calibration a mixture of DDT and Endrin was analyzed. Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval on 10-16-22 and 10-17-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. However, few compounds exceeded maximum percent difference in the closing daily standard in channel B as summarized in the table below:

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-17-22) %
		Channel B
Heptachlor Epoxide	≤ 20%	29*
Gamma Chlordane	≤ 20%	30*
Alpha Chlordane	≤ 20%	35*
Endosulfan I	≤ 20%	30*

The exceedances were all high biased and none of these compounds were detected in the field samples. In the three continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD and MS/MSD. Sample TMW63102022 (Lab ID#J051-08) was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within established QC limits.

Results and recoveries of QC samples were confirmed with the reported raw data.

Results for method blank were reviewed for each component and no organochlorine pesticide was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Field sample TMW51102022 was identified as field duplicate of sample TMW51102022D. No pesticide was detected in the sample and associated field duplicate sample.

4.3.6. Raw data was submitted for all samples. Sample MW29102022(Lab ID#22J051-01) was selected as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. Polychlorinated Biphenyls (EPA Method 3520C/ 8082A)

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Six water samples were collected on 10-05-22, extracted on 10-10-22, and analyzed on 10-13-22, within holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 09-07-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

Retention time window width were established at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 09-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-13-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD and MS/MSD. Sample TMW63102022 (Lab ID#J051-08) was designated to be spiked as MS/MSD. Results and recoveries of LCS/LCSD and MS/MSD was used for both accuracy and precision. Percent recoveries (%R) were within the established QC limits.

Results for method blank was reviewed for each component and no target analyte was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample: Field sample TMW51102022 was identified as field duplicate of sample TMW51102022D. No Polychlorinated Biphenyls was detected in the sample and associated field duplicate sample.

4.4.6. Raw data was submitted for all samples. Sample MW29102022(Lab ID#22J051-01) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Six water samples were collected on 10-05-22, extracted on 10-11-22 and analyzed on 10-13-22 and 10-14-22.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 10-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99819 was used for MCPP in column B.

Retention time windows were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 10-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-13-22 and 10-14-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels.

Results for surrogate recoveries and QC were all reported from channel A. Channel B was used for confirmation only.

4.5.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63102022 (Lab ID#J051-08) was designated to be spiked as MS/MSD. All herbicides target list was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were all within established QC limits

Results for method blank was reviewed for each component and no Herbicide was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.5.5. Field duplicate sample and its associated sample: Field sample TMW51102022 was identified as field duplicate of sample TMW51102022D. No Herbicide was detected in the sample and associated field duplicate sample.

4.5.6. Raw data was submitted for all samples. Sample MW29102022(Lab ID#22J051-01) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Nitroaromatics by HPLC/UV (EPA Method 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for ten (10) water samples requested for this method. Water samples were collected on 10-05-22, extracted on 10-10-22 and analyzed on 10-12-22, within holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis. Positive results, if any, were confirmed with UHPLC equipped with different column (Kinetex- Biphenyl column).

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-25-22 and 08-26-22. Confirmation (Kinetex-Biphenyl column) was calibrated on 08-23-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

Retention time windows were established for each target analyte at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for both primary and confirmation columns on 08-23-21 and 08-26-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of four continuing calibration standards were analyzed on 10-12-22, bracketing the analyses of samples and all the QC samples. Three daily standards were also analyzed with confirmation column on 10-17-22. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63102021 (Lab ID#J051-08) was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.6.5 Field duplicate sample and its associated sample**:** Sample TMW51102022 was identified as field duplicate of sample TMW51102022D. No explosive target compound was detected in each sample and field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample TMW07102022(Lab ID#J051-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation (Biphenyl column).

4.7. Nitroglycerine and PETN by UHPLC/UV (EPA Method 8332)

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for ten (10) water samples requested for this method. Water samples were collected on

10-05-22, extracted on 10-10-22 and analyzed on 10-10-22.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis.

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 08-24-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time windows were established for each target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 08-24-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of six continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-10-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63102021 (Lab ID#J051-08) was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Sample TMW51102022 was identified as field duplicate of sample TMW51102022D. No explosive target compound was detected in sample and field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample TMW07102022(Lab ID#J051-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.8. Total Petroleum hydrocarbons GRO (EPA Method 8015G)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for nine water samples requested for this method. Water samples were collected on 10-05-22. Samples were analyzed on 10-13-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Sample was carried through the system by purge and trap.

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-06-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10). Retention times for further sample analyses was used for peak identification and integration range.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-06-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-12-22 and 10-13-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63102022 (Lab ID#J051-08) was designated to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.8.5. Field duplicate sample and its associated sample: Field sample TMW51102022 was identified as field duplicate of sample TMW51102022D. No TPH as Gasoline was detected in the sample and associated field duplicate sample.

4.8.6. Raw data was submitted for all samples. Sample TMW07102022(Lab ID#22J051-02) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight water sample requested for this method. Water samples were collected on 10-05-22, extracted on 10-10-22 and analyzed on 10-12-22.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Heavier range of total petroleum hydrocarbons were extracted and introduced into system by direct injection.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-08-22. Calibration factor (area for each compound/concentration) was used to quantify diesel range hydrocarbons (TPH as DRO). A second set of initial calibration curve was generated for lighter TPHs (Jet Fuel) and heavier TPHs (motor oil). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time window width was established by analysis of a window defining hydrocarbon standard (C10-C40). Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-08-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

A total of four continuing calibration standards were analyzed on 10-12-22 at 10-injections interval, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63102022 (Lab ID#J051-08) was designated for MS/MSD. Percent recoveries (%R) of LCS/LCSD and MS/MSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.9.5. Field duplicate sample and its associated sample: Field sample MW51102022 was identified as field duplicate of sample TMW51102022D. No TPH as Diesel was detected in the sample and associated field duplicate sample.

4.9.6. Raw data was submitted for all samples. Sample TMW07102022(Lab ID#22J051-02) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.10. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. eleven (11) water samples were collected on 10-04-22 and 10-05-22. Samples were analyzed on 10-06-22 and 10-07-22 within required holding time.

A High-Performance LC coupled with Mass Detector (HPLC/MS) was used for analysis.

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-21-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9960 (Perchlorate ion 83) and 0.9987 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

Retention time for each isotope (ion 83 and 85) at each calibration level was within 0.2 minutes required by the method.

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-21-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of seven daily standards were carried out on 10-06-22 and 10-07-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and two sets of MS/MSD. Samples TMW63102022 (Lab ID#J051-08) and TMW27102022 (Lab ID #J051-12) were designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets MS/MSD and LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. No surrogate is used in this method.

4.10.5. Field duplicate sample and its associated sample: Sample TMW51102022 was identified as field duplicate of TMW51102022D. No perchlorate was detected in sample and associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample MW29102022(Lab ID#J051-01) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of water samples requested for this method. A total of eleven (11) water samples were collected on 10-05-22. Samples were analyzed on 10-06-22, 10-07-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Samples were re-analyzed on 10-24-22, 10-25-22 and 10-26-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

4.11.2. 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 05-04-22 and 10-18-22. 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 05-04-22 and 10-18-22. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-06-22, 10-07-22, 10-24-22, 10-25-22 and 10-26-22. A total of fourteen 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 the 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.

4.11.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD, MS/MSD and sample/sample duplicate. Sample TMW63102022 was designated to be spiked as MS/MSD and sample/sample duplicate. Two more sets of MB and LCS/LCSD was analyzed with Chloride and Sulfate analysis on 10-24-22 and 10-25-22. 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.

Recoveries of MS/MSD were within acceptable limits except for the anions listed in the table below:

Anion	TMW63102022 MS%	TMW63102022 MSD%	% Acceptance limits
Bromide	60*	58*	91-110
Nitrate	86*	79*	88-111
Nitrite	135*	132*	87-111
O-Phosphate	80	65*	80-116
Chloride	120*	114*	87-111

*Outside Acceptance limits

4.11.4. Field duplicate sample and its associated sample: Sample TMW51102022 was identified as field duplicate of TMW51102022D. Results for sample/sample duplicate is summarized in the table below:

Anion	TMW51102022 (Lab ID #J051-10) mg/L	TMW51102022D (Lab ID #J051-11) mg/L	**% RPD**
Nitrate	5.3	5.1	3.85
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.80	0.80	<1
Chloride	140	130	7.41
Fluoride	0.41	0.41	<1
Sulfate	1000	1100	9.52

4.11.5. Raw data was submitted for all requested field samples. Samples TMW07102022 (Lab ID#22J051-02) was designated 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 #22J051 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. 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.

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J086 Analytical Data Package

Publication Date: 02-23-2023

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 2022 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 seven (7) water samples were collected on 10-06-22. EMAX Laboratories received the samples on 10-07-22. 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 MW03102022 (Lab ID#J086-05) was designated 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. Sample TMW01102022 was designated to be spiked as MS/MSD. Recoveries and results of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision. Raw data for method blanks, MS/MSD and LCS/LCSDs were also 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 (7 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 # 22J086) 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 seven (7) water samples were collected on 10-06-22. EMAX Laboratories received the samples on 10-07-22.

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 MW03102022 was designated 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. Sample TMW01102022 was designated to be spiked as MS/MSD on the chain of custody. Raw data for MS/MSD with each set of 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#22J086 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
MW31S102022	J086-01	10-06-22	S3VM	Anions by IC,
TMW10102022	J086-02	10-06-22	S3VM	Anions by IC,
TMW22102022	J086-03	10-06-22	S3VM	Anions by IC,
TMW29102022	J086-04	10-06-22	S3VM	Anions by IC,
MW03102022	J086-05	10-06-22	S3VM	Anions by IC,
MW03102022D	J086-06	10-06-22	S3VM	Anions by IC,
TMW01102022	J086-07	10-06-22	S3VM	Anions by IC,
TMW01102022MS	J086-07M	10-06-22	S3VM	Anions by IC,
TMW01102022MSD	J086-07S	10-06-22	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
		Analysis within 28 days for Bromide, Fluoride, Chloride and Sulfate	None. Holding times were met

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 4.7˚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:

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

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) =7/7X100=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 seven 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 seven (7) water samples were collected on 10-06-22. Samples were analyzed on 10-07-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Samples were re-analyzed on 10-24-22 and 10-25-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

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 05-04-22 and 10-18-22. 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 05-04-22 and 10-18-22. Recoveries were all within

90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-07-22, 10-24-22, and 10-25-22. A total of ten 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, one set of LCS/LCSD, MS/MSD and sample/sample duplicate. Sample TMW01102022 was designated to be spiked as MS/MSD and sample/sample duplicate. One more set of MB and LCS/LCSD was analyzed with Chloride and Sulfate analysis on 10-24-22. 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.

Recoveries of MS/MSD were mostly within acceptable limits except for few anions as indicated in the table below:

Anion	TMW01102022 (Lab ID#J086-07) MS%	TMW01102022 (Lab ID#J086-07) MSD%	% Acceptance limits
Bromide	114*	111*	91-110
Nitrate	88	87*	88-111
Nitrite	112*	111	87-111

*Outside Acceptance limits

4.1.4. Field duplicate sample and its associated sample: Sample MW03102022 was identified as field duplicate of MW03102022D. Results for sample/sample duplicate is summarized in the table below:

Anion	MW03102022 (Lab ID #J086-05) mg/L	MW03102022D (Lab ID #J086-06) mg/L	**% RPD**
Nitrate	5.3	5.3	<1
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.36J	0.51	34.5
Chloride	270	280	3.64
Fluoride	0.30	0.46	42.1
Sulfate	1500	1400	2.60

4.1.5. Raw data was submitted for all requested field samples. Sample MW03102022 (Lab ID#22J086-05) was designated 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 #22J086 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.

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J122 Analytical Data Package

Publication Date: 02-16-23

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 2022 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 sixteen (16) water samples were collected on 10-07-22. EMAX Laboratories received the samples on 10-08-22. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample MW30102022(Lab ID# J122-02) was designated as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation review. No sample was designated to be spiked as MS/MSD on the chain of custody. Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision. Raw data for method blank and LCS/LCSD for each method 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, (both extraction and analysis), extraction logs, 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 methods of analysis:

EPA Method 3050B/8260C: Volatile Organics by GC-MS (16 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (11 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (13 samples)
EPA Method SW8332: Nitroglycerine and PETN (13 samples)
EPA Method 8081B: Organochlorine Pesticides (10 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (7 sample)

EPA Method 8151A: Chlorinated herbicides (7 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (9 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (8 samples)
EPA Method 6850: Perchlorate (13 samples)
EPA Method 9056A: Anions by IC (15 samples)

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 Methods (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 extraction and analytical holding times were met for each method and all the related samples. The deviations, if any, are discussed in Section 4.0 for each method.

The analytical data evaluated in this data validation report (SDG # 22J122) has met the data quality and usability requirement as defined in the data quality objectives. While very few analytical QC exceedances were observed, it was not significant for any data qualifiers. 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 DoD *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 sixteen (16) water samples were collected on 10-07-22. EMAX Laboratories received the samples on 10-08-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the 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 preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), when applicable
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses 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. Next, selected analytical reports including QA/QC information was cross checked with raw data. The analysis and extraction sequence logs for each method were 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, surrogate recoveries, MS/MSD, and lab control samples (LCS/LCSD) 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 each analytical method requested on the chain of custody were submitted for all samples. One field sample MW30102022 (Lab ID# J122-02) from this sample delivery group was designated as stage 3 data deliverable on the chain of custody. Raw data for this sample was evaluated comprehensively. No sample was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank and LCS/LCSD was reviewed in detail. 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 regard to the requested method of analysis (collection to extraction and extraction 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
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated;
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards, and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

The following is a list of sample identifications and corresponding laboratory sample identification numbers:

Site Name: Fort Wingate, New Mexico

SDG#22J122 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW23102022	22J122-01	10-07-22	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC
MW30102022	22J122-02	10-07-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides Anions by IC
TMW33102022	22J122-03	10-07-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 TPH Gasoline; TPH as DRO Anions by IC
TMW41102022	22J122-04	10-07-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC
MW27102022	22J122-05	10-07-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides Anions by IC
TMW02102022	22J122-06	10-07-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22J122 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW02102022D	22J122-07	10-07-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC
TMW55102022	22J122-08	10-07-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides Anions by IC
MW31102022	22J122-09	10-07-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides Anions by IC
TMW39S102022	22J123-10	10-07-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC
TMW62102022	22J122-11	10-07-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22J122 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW04102022	22J122-12	10-07-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 Anions by IC
TMW64102022	22J122-13	10-07-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides Anions by IC
TMW28102022	22J122-14	10-07-22	S3VM	VOCs by SW5030B/8260C, Anions by IC
QC07102022 EB3	22J122-15	10-07-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides Anions by IC
QC07102022 TB5	22J122-16	10-07-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8330B	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in seventeen ice preserved coolers.

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction, and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements 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"
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	NA
Anions by IC	Water	Analysis 48 hours from collection for Nitrate, Nitrite &Orthophosphate and 28-days for Bromide, Chloride, Fluoride and Sulfate	Holding times were met for all
Mercury & Dissolved Mercury	Water	Collection to Analysis: 28 days	NA

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 and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in seventeen ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 0.8˚C and as high as 4.8˚C upon arrival. All samples were received intact and 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 exactly the same way. 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. The completeness objective is to obtain a sufficient amount 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 5030B/8260C: VOCs) =16/16X100=100%

Completeness (EPA Method 3520B/8270D: SVOCs) =11/11X100=100%

Completeness (EPA Method 8330B: Explosives) =13/13X100=100%

Completeness (EPA Method 8332: Nitroglycerine & PETN) =13/13X100=100%

Completeness (EPA Method 8081B: Organochlorine pesticides) =10/10X100=100%

Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =7/7X100=100%

Completeness (EPA Method 8151B: Chlorinated Herbicides) =7/7X100=100%

Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 9/9X100=100%

Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =8/8X100=100%

Completeness (EPA Method 6850: Perchlorate) =13/13X100=100%

Completeness (EPA Method 9056A: Anions) =15/15X100=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 sixteen water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
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. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Sixteen water samples were collected on 10-07-22. Samples were analyzed on 10-21-22 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

Chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.1.7.

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to each set of initial calibration on 10-15-22, 10-17-22 and at the beginning of each analysis shift on 10-21-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to two sets of initial calibration using two different GC/MSD. Initial calibration curves were generated on 10-15-22 and 10-17-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table:

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 10-15-22	Average Response factor 10-17-22
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Bromoform	≥ 0.10	√	√
Chlorobenzene	≥ 0.50	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.045 & 0.036) and 2-Butanone (0.087 & 0.014).

However, recoveries were within the requirement of 70-130%. Calibration check compounds (CCCs) met the acceptance criteria for %RSD among the response factors calculated for each level. Table 4.1.3.2 lists the CCCs with method requirement limits for %RSD among response factors for initial calibration.

Table 4.1.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Response Factors %RSD 10-15-22	Response Factors %RSD 10-17-22
Vinyl chloride	-≤ 20%	√	√
1,1-Dichloroethene	-≤ 20%	√	√
Chloroform	-≤ 20%	√	√
1,2-Dichloropropane	-≤ 20%	√	√
Toluene	-≤ 20%	√	√
Ethyl benzene	-≤ 20%	√	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard on 10-15-22 and 10-17-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standard was analyzed at the beginning and end of each analysis shift on 10-21-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-21-22 (I&II)	Continuing cal. Response factors 10-21-22 (I&II)	Continuing cal. Response factors 10-21-22 (I&II)
		1st Analysis shift	2nd Analysis shift	3rd Analysis shift
Chloromethane	≥ 0.10	√	√	√
1,1-Dichloroethane	≥ 0.20	√	√	√
Chlorobenzene	≥ 0.50	√	√	√

System Performance Check compounds (SPCCs)	**Minimum response factor (Method limits)**	**Continuing cal. Response factors 10-21-22 (I&II)**	**Continuing cal. Response factors 10-21-22 (I&II)**	**Continuing cal. Response factors 10-21-22 (I&II)**
		1st Analysis shift	2nd Analysis shift	3rd Analysis shift
Bromoform	≥ 0.10	√	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. The calculated % difference between RFs from continuing calibration and average response factors from initial calibration is summarized in Table 4.1.4.2 for continuing calibration reports presented with the data package.

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	**%Deviation From Initial calibration (Acceptance Limit)**	**%Deviation from Initial calibration (10-21-22)** I&II	**%Deviation from Initial calibration (10-21-22)** I&II	**%Deviation from Initial calibration (10-21-22) I&II**
		1st Analysis shift	2nd Analysis shift	3rd Analysis shift
Vinyl chloride	≤ 20%	√	√	√
1,1-Dichloroethene	≤ 20%	√	√	√
Chloroform	≤ 20%	√	√	√
1,2-Dichloropropane	≤ 20%	√	√	√
Toluene	≤ 20%	√	√	√
Ethyl benzene	≤ 20%	√	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list except for compounds listed in the table below for each daily standard:

Target Compounds	**%Deviation From In. calibration (Accept. Limit)**	**%Deviation from Initial calibration (10-21-22)** 1st Analysis shift		**%Deviation from Initial calibration (10-21-22)** 2nd Analysis shift		**%Deviation from Initial calibration (10-21-22)** 3rd Analysis shift	
		opening	closing	opening	closing	opening	closing
2-Chloroethylvinyl ether	≤ 20%	--	--	--	--	34.3*	24.8*
1,2-Dibromochloropropane	≤ 20%	21.8*	--	21.8*	--	--	--
4-Chlorotoluene	≤ 20%	--	21.1*	--	--	--	--
2-Chlorotoluene	≤ 20%	--	--	--	23.9*	--	--
Hexachlorobutadiene	≤ 20%	--	--	--	--	--	22.7*

*Outside control limits; I: Opening daily standard; II: Closing daily standard

These compounds were not detected in the corresponding field samples. This should not affect the data quality.

4.1.5. Quality Control samples reported consisted of three method blanks and three sets of LCS/LCSD. No sample was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blanks presented with the data package, analyzed with samples did not show presence of any target compounds.
Surrogate recoveries were all within the method's acceptable limits.

4.1.6. **Field duplicate sample** and its associated sample: Field sample TMW02102022 was identified as field duplicate of sample TMW02102022D. No Volatile Organic Compounds (VOCs) were detected in sample or field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample MW30102022 (Lab ID# 22J122-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

4.2. SVOC (EPA Method 3520C/8270D)

4.2.1. Technical Holding Times

Holding time requirement was met for all eleven samples. Water samples were collected on 10-07-22, extracted on 10-13-22 and were analyzed on 10-18-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 10-18-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group.

Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table.

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-07-22
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Average response factors for the rest of target compounds were within method's recommended values.

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 20%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-07-22
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 03-07-22
Benzoic acid	0.9969
2,4-Dinitrophenol	0.9964
4-Nitrophenol	0.9971
Di-n-Octyl phthalate	0.9972

All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-18-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (10-18-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for all other target

analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Percent difference between RFs from continuing calibration and average response factors from initial calibration passed the method's criteria as summarized in Table 4.2.4.2.

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (10-18-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds in the opening daily standard as listed in the table below:

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	Deviation from Initial calibration (10-18-22) %	
		opening	closing
2,4-Dinitrophenol	≤ 20%	28.2*	--
1,3-Dinitrobenzene	≤ 20%	22.2*	--

*Outside control limits

The exceedances were high biased and the compounds were not detected in the corresponding field samples. This should not affect the data quality.

4.2.5. Quality Control samples reported consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. The full list of target

compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for the QC samples reported were all within the project acceptance limits.
Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision in this method.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.
Surrogate recoveries were all within the method's acceptable limits.

4.2.6. **Field duplicate sample** and its associated sample: No field duplicate was assigned to this method.

4.2.7. Raw data was submitted for all samples. Sample MW30102022(Lab ID#J122-02) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.3. ORGANOCHLORINE PESTICIDES (EPA Method 3520C/ 8081B)

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for ten water samples requested for this method. Water samples were collected on 10-07-22, extracted on 10-13-22 and analyzed on 10-14-22 and 10-15-22 within required holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 09-02-22, 09-03-22 and before sample analysis on 10-14-22 and 10-15-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 09-02-22 and 09-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 08-11-22 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene.

Retention time window width were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 08-11-22 and 09-12-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Performance of instrument was monitored by analysis of DDT and Endrin breakdown mixture. Before each continuing (daily) calibration a mixture of DDT and Endrin was analyzed. Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval on 10-14-22 and 10-15-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. In the three continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) and %RPDs were within established QC limits. Results and recoveries of QC samples were confirmed with the reported raw data.

Results for method blank were reviewed for each component and no organochlorine pesticide was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: No field duplicate sample was collected for this method.

4.3.6. Raw data was submitted for sample and all QC samples. Sample MW30102022 (Lab ID#22J122-02) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. Polychlorinated Biphenyls (EPA Method 3520C/ 8082A)

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Seven water samples were collected on 10-07-22, extracted on 10-13-22, and analyzed on 10-14-22 and 10-15-22, within holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 09-07-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

Retention time window width were established at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 09-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-14-22 and 10-15-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Results and recoveries of LCS/LCSD was used for both accuracy and precision. Percent recoveries (%R) were within the established QC limits.

Results for method blank was reviewed for each component and no target analyte was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample: No field duplicate sample was assigned to this method.

4.4.6. Raw data was submitted for all samples. Sample MW30102022 (Lab ID#J122-02) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Seven water samples were collected on 10-07-22, extracted on 10-14-22 and analyzed on 10-17-22 and 10-18-22.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 10-03-22 and 10-04-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99819 was used for MCPP in column B.

Retention time windows were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 10-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-17-22 and 10-18-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% except for the compounds summarized in the table below in the closing daily standard:

Target Compounds	**%Deviation From Initial calibration (Accept. Limit)**	**%Deviation from Initial calibration (10-18-22) Closing daily std.**	**%Deviation from Initial calibration (10-18-22) Closing daily std.**
		Channel A	Channel B
Acifluorfen	≤ 20%	23*	33*
Dinoseb	≤ 20%	--	26*

*Outside QC limit

None of these compounds were detected in the field samples, and it should not affect the quality of data.

Results for surrogate recoveries and QC were all reported from channel A. Channel B was used for confirmation only.

4.5.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Full herbicides target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits

Results for method blank was reviewed for each component and no Herbicide was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.5.5. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this method.

4.5.6. Raw data was submitted for all samples. Sample MW30102022(Lab ID#J122-02) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Nitroaromatics by HPLC/UV (EPA Method 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen (13) water samples requested for this method. Water samples were collected on 10-07-22, extracted on 10-13-22 and analyzed on 10-14-22 and 10-15-22, within holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis. Positive results, if any, were confirmed with UHPLC equipped with different column (Kinetex- Biphenyl column).

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-25-22 and 08-26-22. Confirmation (Kinetex-Biphenyl column) was calibrated on 08-23-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

Retention time windows were established for each target analyte at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for both columns on 08-23-21 and 08-26-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of four continuing calibration standards were analyzed on 10-14-22 and 10-15-22, bracketing the analyses of samples and all the QC samples. Three more daily standards were analyzed with confirmation column on 10-17-22. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Full explosive target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.6.5 Field duplicate sample and its associated sample**:** Field sample TMW02102022 was identified as field duplicate of sample TMW02102022D. Traces of 3,5-Dinitroaniline was detected in the sample and associated field duplicate sample as summarized in the table below:

Explosives	TMW02102022 (Lab ID #J122-06) µg/L	TMW02102022D (Lab ID #J122-07) µg/L	**% RPD**
3,5-Dinitroaniline	0.21J	0.19J	10.0

4.6.6. Raw data was submitted for all samples. Sample MW30102022(Lab ID#J122-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation column (Biphenyl column).

4.7. Nitroglycerine and PETN by UHPLC/UV (EPA Method 8332)

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen (13) water samples requested for this method. Water samples were collected on 10-07-22, extracted on 10-13-22 and analyzed on 10-14-22 within the holding time requirements. A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis.

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 08-24-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time windows were established for each target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 08-24-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-14-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Field sample TMW02102022 was identified as field duplicate of sample TMW02102022D. No explosive was detected in the sample and associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample MW30102022(Lab ID#J122-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.8. Total Petroleum hydrocarbons GRO (EPA Method 8015G)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for nine water samples requested for this method. Water samples were collected on 10-07-22. Samples were analyzed on 10-16-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Sample was carried through the system by purge and trap.

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-06-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10). Retention times for further sample analyses was used for peak identification and integration range.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-06-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-16-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample MW31102022 (Lab ID#J122-09) was selected to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD and MS/MSD.

Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.8.5. Field duplicate sample and its associated sample: No field duplicate of sample was assigned to this method.

4.8.6. Raw data was submitted for all samples. Sample MW30102022(Lab ID#J122-02) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight water sample requested for this method. Water samples were collected on 10-07-22, extracted on 10-14-22 and analyzed on 10-18-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Heavier range of total petroleum hydrocarbons were extracted and introduced into system by direct injection.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-08-22. Calibration factor (area for each compound/concentration) was used to quantify diesel range hydrocarbons (TPH as DRO). A second set of initial calibration curve was generated for lighter TPHs (Jet Fuel) and heavier TPHs (motor oil). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time window width was established by analysis of a window defining hydrocarbon standard (C10-C40). Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-08-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

A total of three continuing calibration standards were analyzed on 10-18-22 at 10-injections interval, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated for MS/MSD. Percent recoveries (%R) of LCS/LCSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.9.5. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this method.

4.9.6. Raw data was submitted for all samples. Sample MW30102022(Lab ID#J122-02) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.10. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen water samples requested for this method. Water samples were collected on 10-07-22. Samples were analyzed on 10-10-22 and 10-11-22 within required holding time.

A High-Performance LC coupled with Mass Detector (HPLC/MS) was used for analysis.

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-21-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9960 (Perchlorate ion 83) and 0.9987 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

Retention time for each isotope (ion 83 and 85) at each calibration level was within 0.2 minutes required by the method.

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-21-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of seven daily standards were carried out on 10-10-22 and 10-11-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established QC limits for both sets MS/MSD and LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. No surrogate is used in this method.

4.10.5. Field duplicate sample and its associated sample: Field sample TMW02102022 was identified as field duplicate of sample TMW02102022D. Perchlorate results for sample and associated field duplicate sample is summarized in the table below:

Perchlorate	TMW02102022 (Lab ID #J122-06) µg/L	TMW02102022D (Lab ID #J122-07) µg/L	**% RPD**
Perchlorate_83	6.3	6.3	≤1

4.10.6. Raw data was submitted for all samples. Sample MW30102022(Lab ID#J122-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of water samples requested for this method. A total of fifteen (15) water samples were collected on 10-07-22. Samples were analyzed on 10-08-22 and 10-09-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Samples were re-analyzed with dilution on 10-25-22 and 10-26-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

4.11.2. 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 05-04-22 and 10-18-22. 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 05-04-22 and 10-18-22. Recoveries were all within

90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-08-22, 10-09-22, 10-25-22 and 10-26-22. A total of ten 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 the 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.

4.11.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD, MS/MSD and sample/sample duplicate. Sample TMW41102022 (Lab ID#J122-04) was selected to be spiked as MS/MSD and sample/sample duplicate. One more set of MB and LCS/LCSD was analyzed with Chloride and Sulfate re-analysis on 10-25-22. 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 and MS/MSD for all anions. Recoveries of MS/MSD were within acceptable limits except for the anions listed in the table below:

Anion	TMW41102022 (Lab ID#J122-04) MS%	TMW41102022 (Lab ID#J122-04) MSD%	% Acceptance limits
Bromide	61*	60*	91-110
Nitrate	87*	86*	88-111
Nitrite	130*	131*	87-111
Sulfate	80*	76*	87-112
Chloride	99	116*	87-111

*Outside Acceptance limits

Therefore, the positive results in the parent sample will be flagged as estimated value “J” and non-detect results as “UJ”.

4.11.4. Field duplicate sample and its associated sample: Sample TMW02102022 was identified as field duplicate of TMW02102022D. Results for sample/sample duplicate is summarized in the table below:

Anion	TMW02102022 (Lab ID #J122-06) mg/L	TMW02102022D (Lab ID #J122-07) mg/L	**% RPD**
Nitrate	60	60	<1
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	1.3	1.3	<1
Chloride	290	320	9.84
Fluoride	0.28	0.28	<1
Sulfate	1200	1300	8.0

4.11.5. Raw data was submitted for all requested field samples. Sample MW30102022 (Lab ID#22J122-02) was designated 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 #22J122 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. 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.

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J123 Analytical Data Package

Publication Date: 02-15-23

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 2022 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 eight (8) water samples were collected on 10-06-22. EMAX Laboratories received the samples on 10-08-22. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample MW03102022(Lab ID# J123-05) was designated as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation review. Sample TMW01102022 (Lab ID# J123-07) was designated to be spiked as MS/MSD on the chain of custody. Results and recoveries of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision. Raw data for method blank, LCS/LCSD and MS/MSD for each method 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, (both extraction and analysis), extraction logs, 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 methods of analysis:

EPA Method 3050B/8260C: Volatile Organics by GC-MS (8 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (2 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (7 samples)
EPA Method SW8332: Nitroglycerine and PETN (7 samples)
EPA Method 8081B: Organochlorine Pesticides (1 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (4 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (3 samples)
EPA Method 6850: Perchlorate (7 samples)

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 Methods (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 extraction and analytical holding times were met for each method and all the related samples. The deviations, if any, are discussed in Section 4.0 for each method.

The SDG # 22J123 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. While very few analytical QC exceedances were observed, it was not significant for any data qualifiers. 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 DoD *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 eight (8) water samples were collected on 10-06-22. EMAX Laboratories received the samples on 10-08-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the 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 preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD),
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses 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. Next, selected analytical reports including QA/QC information was cross checked with raw data. The analysis and extraction sequence logs for each method were 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, surrogate recoveries, MS/MSD, and lab control samples (LCS/LCSD) 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 each analytical method requested on the chain of custody were submitted for all samples. One field sample MW03102022 (Lab ID# J123-05) from this sample delivery group was designated as stage 3 data deliverable on the chain of custody. Raw data for this sample was evaluated comprehensively. Sample TMW01102022 was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank, LCS/LCSD and MS/MSD was reviewed in detail. 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 regard to the requested method of analysis (collection to extraction and extraction 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
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated;
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards, and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

The following is a list of sample identifications and corresponding laboratory sample identification numbers:

Site Name: Fort Wingate, New Mexico

SDG#22J123 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW31S102022	22J123-01	10-06-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
TMW10102022	22J123-02	10-06-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline; TPH as DRO Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
TMW22102022	22J123-03	10-06-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
TMW29102022	22J123-04	10-06-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
MW03102022	22J123-05	10-06-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
MW03102022D	22J123-06	10-06-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
TMW01102022	22J123-07	10-06-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#22123 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
QC06102022 TB4	22J123-08	10-06-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW01102022MS	22J123-07M	10-06-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
TMW01102022MSD	22J123-07S	10-06-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850

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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8330B	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in six ice preserved coolers.

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction, and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements 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"
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	NA
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	NA
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	NA
Anions by IC	Water	Analysis 48 hours from collection for Nitrate, Nitrite &Orthophosphate and 28-days for Bromide, Chloride, Fluoride and Sulfate	NA
Mercury & Dissolved Mercury	Water	Collection to Analysis: 28 days	NA

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 and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in six ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.1˚C and as high as 1.9˚C upon arrival. All samples were received intact and 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 exactly the same way. 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. The completeness objective is to obtain a sufficient amount 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 5030B/8260C: VOCs) =8/8X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =2/2X100=100%
Completeness (EPA Method 8330B: Explosives) =7/7X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =7/7X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =1/1X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 4/4X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =3/3X100=100%
Completeness (EPA Method 6850: Perchlorate) =7/7X100=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 eight water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS

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. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Eight water samples were collected on 10-06-22. Samples were analyzed on 10-20-22 within method's requirement for holding time. (Water samples were preserved with hydrochloric acid).

Chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.1.7.

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 10-15-22 and at the beginning of analysis shift on 10-20-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 10-15-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table:

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 10-15-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.045) and 2-Butanone (0.087).

However, recoveries were within the requirement of 70-130%. Calibration check compounds (CCCs) met the acceptance criteria for %RSD among the response factors calculated for each level. Table 4.1.3.2 lists the CCCs with method requirement limits for %RSD among response factors for initial calibration.

Table 4.1.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Response Factors %RSD 10-15-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 10-15-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standard was analyzed at the beginning and end of analysis shift on 10-20-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-20-22 (Opening)	Continuing cal. Response factors 10-20-22 (closing)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for

all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. The calculated % difference between RFs from continuing calibration and average response factors from initial calibration is summarized in Table 4.1.4.2 for continuing calibration compounds presented with the data package.

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration 10-20-22 (Opening)	%Deviation from Initial calibration 10-20-22 (Closing)
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list except for compounds in the table below for each daily standard:

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-20-22) I %	%Deviation from Initial calibration (10-20-22) II %
2-Chloroethylvinyl ether	≤ 20%	40.0*	√
Chloromethane	≤ 20%	29.5*	20.8*
Acetone	≤ 20%	√	28.9*

*Outside control limits

At least twelve compounds exceeded %D in the closing daily standard, all high biased. These compounds were not detected in the corresponding field samples. This should not affect the data quality.

4.1.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW01102022 (Lab ID# J123-07) was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list, except for one compound in MS/MSD as listed in the table below:

VOC Target Compound	TMW01102022 (J123-07) MS%	TMW01102022 (J123-07) MSD%	**%** Acceptance limits
Bromochloromethane	128*	127*	78-123

This compound was not detected in the corresponding field samples, therefore, would not affect the data quality.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blanks presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method's acceptable limits.

4.1.6. **Field duplicate sample** and its associated sample: Field sample MW03102022 was identified as field duplicate of sample MW03102022D. Traces of Acetone was detected in the sample and associated field duplicate sample as indicated in the table below:

VOC Target Compound	MW03102022 (J123-05)	MW03102022D (J123-06)	**%RP**D
Acetone	5.2J	6.8J	26.7

4.1.7. Raw data was submitted for all samples. Sample MW03102022 (Lab ID# 22J123-05) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

4.2. SVOC (EPA Method 3520C/8270D)

4.2.1. Technical Holding Times

Holding time requirement was met for all nine samples. Water samples were collected on 10-06-22, extracted on 10-13-22 and were analyzed on 10-18-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 10-18-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group.

Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table.

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-07-22
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Average response factors for the rest of target compounds were within method's recommended values.

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 20%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-07-22
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	**Least Square Linear Regression (CCF) 03-07-22**
Benzoic acid	0.9969
2,4-Dinitrophenol	0.9964
4-Nitrophenol	0.9971
Di-n-Octyl phthalate	0.9972

All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-18-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	**Minimum response factor (Method limits)**	**Continuing cal. Response factors (10-18-22) I&II**
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for all other target

analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Percent difference between RFs from continuing calibration and average response factors from initial calibration passed the method's criteria as summarized in Table 4.2.4.2.

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	**% Deviation From Initial calibration (Acceptance Limit)**	**Accepted Deviation from Initial calibration (10-18-22) I& II**
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds from closing daily standard as listed in the table below:

Target Compounds	**%Deviation From Initial calibration (Acceptance Limit)**	**%Deviation from Initial calibration (10-18-22) %**	
		Opening Daily St.	Closing Daily St.
2,4-Dinitrophenol	≤ 20%	28.2*	√
1,3-Dinitrobenzene	≤ 20%	22.2*	√

*Outside control limits

The exceedances were high biased and the compounds were not detected in the corresponding field samples. This should not affect the data quality.

4.2.5. Quality Control samples reported consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for the QC samples reported were all within the project acceptance limits.

Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision in this method.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method's acceptable limits.

4.2.6. **Field duplicate sample** and its associated sample: No field duplicate was assigned to this method.

4.2.7. Raw data was submitted for all samples. Sample TMW31S102022(Lab ID#J123-01) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.3. ORGANOCHLORINE PESTICIDES (EPA Method 3520C/ 8081B)

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for one water sample requested for this method. Water sample was collected on 10-06-22, extracted on 10-12-22 and analyzed on 10-16-22 within required holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 03-11-22, 03-12-22 and before sample analysis on 10-16-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 03-11-22 and 03-12-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 03-12-22 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene.

Retention time window width were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 03-12-22 and 03-15-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Performance of instrument was monitored by analysis of DDT and Endrin breakdown mixture. Before each continuing (daily) calibration a mixture of DDT and Endrin was analyzed. Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval on 10-16-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from both channels A and B. In the three continuing calibration standards, one mid-point concentration of 20-40μg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) and %RPDs were within established QC limits. Results and recoveries of QC samples were confirmed with the reported raw data.

Results for method blank were reviewed for each component and no organochlorine pesticide was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: No field duplicate sample was collected for this method.

4.3.6. Raw data was submitted for sample and all QC samples. Sample TMW31S102022(Lab ID#22J123-01) was selected as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. Nitroaromatics by HPLC/UV (EPA Method 8330B)

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven (7) water samples requested for this method. Water samples were collected on 10-06-22, extracted on 10-12-22 and analyzed on 10-12-22 and 10-13-22, within holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis. Positive results, if any, were confirmed with UHPLC equipped with different column (Kinetex- Biphenyl column).

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-25-22 and 08-26-22. Confirmation (Kinetex-Biphenyl column) was calibrated on 08-23-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

Retention time windows were established for each target analyte at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for both columns on 08-23-21 and 08-26-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of five continuing calibration standards were analyzed on 10-12-22 and 10-13-22, bracketing the analyses of samples and all the QC samples. Two daily standards were also analyzed with confirmation column on 10-17-22. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.4.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW01102021 (Lab ID#J123-07) was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.4.5 Field duplicate sample and its associated sample**:** Field sample MW03102022 was identified as field duplicate of sample MW03102022D. No explosives were detected in the sample and associated field duplicate sample.

4.4.6. Raw data was submitted for all samples. Sample MW03102022(Lab ID#J123-05) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation column (Biphenyl column).

4.5. Nitroglycerine and PETN by UHPLC/UV (EPA Method 8332)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven (7) water samples requested for this method. Water samples were collected on 10-06-22, extracted on 10-12-22 and analyzed on 10-14-22 within the holding time requirements.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis.

4.5.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 08-24-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time windows were established for each target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 08-24-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-14-22, bracketing the analyses of samples and all the QC samples.

Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.5.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW01102021 (Lab ID#J123-07) was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.5.5. Field duplicate sample and its associated sample**:** Field sample MW03102022 was identified as field duplicate of sample MW03102022D. No explosive was detected in the sample and associated field duplicate sample.

4.5.6. Raw data was submitted for all samples. Sample MW03102022(Lab ID#J123-05) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Total Petroleum hydrocarbons GRO (EPA Method 8015G)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for four water samples requested for this method. Water samples were collected on 10-06-22. Samples were analyzed on 10-14-22, 10-15-22, 10-17-22 and 10-18-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Sample was carried through the system by purge and trap.

4.6.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-06-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10). Retention times for further sample analyses was used for peak identification and integration range.

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-06-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Eight continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-14-22, 10-15-22, 10-16-22 and 10-18-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.6.4. Quality Control samples consisted of two method blanks and two sets of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank.
Surrogate recoveries were all within the method QC acceptance limits.

4.6.5. Field duplicate sample and its associated sample: Field sample MW03102022 was identified as field duplicate of sample MW03102022D. No TPH as Gasoline was detected in the sample and associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample MW03102022(Lab ID#J123-05) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.7. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for three water sample requested for this method. Water samples were collected on 10-06-22, extracted on 10-13-22 and analyzed on 10-15-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Heavier range of total petroleum hydrocarbons were extracted and introduced into system by direct injection.

4.7.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-08-22. Calibration factor (area for each compound/concentration) was used to quantify diesel range hydrocarbons (TPH as DRO). A second set of initial calibration curve was generated for lighter TPHs (Jet Fuel) and heavier TPHs (motor oil). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time window width was established by analysis of a window defining hydrocarbon standard (C10-C40). Retention times for further sample analyses was used for peak identification and integration range.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-08-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

A total of three continuing calibration standards were analyzed on 10-15-22 at

10-injections interval, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.7.4. **Quality Control** samples consisted of one method blank and one set of LCS/LCSD. No sample was designated for MS/MSD. Percent recoveries (%R) of LCS/LCSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.7.5. **Field duplicate sample** and its associated sample: Field sample MW03102022 was identified as field duplicate of sample MW03102022D. No TPH as DRO was detected in the sample and associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample MW03102022(Lab ID#J123-05) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.8. Perchlorate by HPLC/MS (EPA Method 6850)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven water samples requested for this method. Water samples were collected on 10-06-22. Samples were analyzed on 10-10-22 and 10-11-22 within required holding time.

A High-Performance LC coupled with Mass Detector (HPLC/MS) was used for analysis.

4.8.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-21-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9960 (Perchlorate ion 83) and 0.9987 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

Retention time for each isotope (ion 83 and 85) at each calibration level was within 0.2 minutes required by the method.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-21-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of seven daily standards were carried out on 10-10-22 and 10-11-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.8.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and one set of MS/MSD. Sample TMW01102022 (Lab ID#J123-07) was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets MS/MSD and LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. No surrogate is used in this method.

4.8.5. Field duplicate sample and its associated sample: Field sample MW03102022 was identified as field duplicate of sample MW03102022D. No Perchlorate was detected in the sample and associated field duplicate sample.

4.8.6. Raw data was submitted for all samples. Sample MW03102022(Lab ID#J123-05) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #22J123 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. 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).

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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.

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

Project No: Eco-18-1237

SDG #22J135 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 2022 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 sixteen (16) water samples were collected on 10-10-22. EMAX Laboratories received the samples on 10-11-22. 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 TMW53102022 (Lab ID#J135-04) was designated 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. Sample TMW17102022(Lab ID#J135-09) was designated to be spiked as MS/MSD. Recoveries and results of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision. Raw data for method blanks MS/MSD and LCS/LCSDs were also 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 (16 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 # 22J135) 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 sixteen (16) water samples were collected on 10-10-22. EMAX Laboratories received the samples on 10-11-22.

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,
- 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 TMW53102022 was designated 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. Sample TMW17102022 was designated to be spiked as MS/MSD on the chain of custody. Raw data for MS/MSD with each set of 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, 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#22J135 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
MW33102022	J135-01	10-10-22	S3VM	Anions by IC,
TMW52102022	J135-02	10-10-22	S3VM	Anions by IC,
MW34102022	J135-03	10-10-22	S3VM	Anions by IC,
TMW53102022	J135-04	10-10-22	S3VM	Anions by IC,
TMW14A102022	J135-05	10-10-22	S3VM	Anions by IC,
MW26102022	J135-06	10-10-22	S3VM	Anions by IC,
MW38102022	J135-07	10-10-22	S3VM	Anions by IC,
TMW59102022	J135-08	10-10-22	S3VM	Anions by IC,
TMW17102022	J135-09	10-10-22	S3VM	Anions by IC,
TMW57102022	J135-10	10-10-22	S3VM	Anions by IC,
MW37102022	J135-11	10-10-22	S3VM	Anions by IC,
MW37102022D	J135-12	10-10-22	S3VM	Anions by IC,
TMW61102022	J135-13	10-10-22	S3VM	Anions by IC,
TMW03102022	J135-14	10-10-22	S3VM	Anions by IC,
FW31102022	J135-15	10-10-22	S3VM	Anions by IC,
QC10102022EB4	J135-16	10-10-22	S3VM	Anions by IC,
TMW17102022MS	J135-09M	10-10-22	S3VM	Anions by IC,
TMW17102022MSD	J135-09S	10-10-22	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
		Analysis within 28 days for Bromide, Fluoride, Chloride and Sulfate	None. Holding times were met

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 1.6˚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:

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

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:

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

Completeness (EPA Method 9056A: Anions) =16/16X100=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 sixteen 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 sixteen (16) water samples were collected on 10-10-22. Samples were analyzed on 10-11-22 and 10-12-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Samples were re-analyzed on 10-26-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

4.12.2. 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 05-04-22 and 10-18-22. 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 05-04-22 and 10-18-22. Recoveries were all within

90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-11-22, 10-12-22, and 10-26-22. A total of eleven 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 the 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.

4.12.3. Quality Control Samples consisted of two method blanks, two sets of LCS/LCSD, MS/MSD and sample/sample duplicate. Sample TMW17102022 was designated to be spiked as MS/MSD and sample/sample duplicate. One more set of MB and LCS/LCSD was analyzed with Chloride and Sulfate analysis on 10-26-22. 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.

Recoveries of MS/MSD were failing for all anions as indicated in the table below:

Anion	TMW17102022 (Lab ID#J135-09) MS%	TMW17102022 (Lab ID#J135-09) MSD%	% Acceptance limits
Bromide	56*	56*	91-110
Fluoride	116*	117*	88-112
Nitrate	73*	73*	88-111
Nitrite	119*	119*	87-111
O-Phosphate	60*	61*	80-116
Chloride	114*	106	87-111
Sulfate	113*	106	87-112

*Outside Acceptance limits

Therefore, the positive results for each anion would be qualified as estimated value, “J” and non-detect results as “UJ” in the parent sample.

4.12.4. Field duplicate sample and its associated sample: Sample MW37102022 was identified as field duplicate of MW37102022D. Results for sample/sample duplicate is summarized in the table below:

Anion	MW37102022 (Lab ID #J135-11) mg/L	MW37102022D (Lab ID #J135-12) mg/L	**% RPD**
Nitrate	U	0.040J	200
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.62	0.53	15.6
Chloride	160	190	17.1
Fluoride	1.4	1.4	<1
Sulfate	430	490	13.0

4.12.5. Raw data was submitted for all requested field samples. Sample TMW53102022 (Lab ID#22J135-04) was designated 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 #22J135 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.

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J151 Analytical Data Package

Publication Date: 02-27-2023

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 2022 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 eleven (11) water samples were collected on 10-11-22. EMAX Laboratories received the samples on 10-12-22. 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 TMW43102022 (Lab ID#J151-01) was designated 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. Sample TMW15102022(Lab ID#J151-04) was designated to be spiked as MS/MSD. Recoveries and results of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision. Raw data for method blanks MS/MSD and LCS/LCSDs were also 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 (11 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 # 22J151) 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 eleven (11) water samples were collected on 10-11-22. EMAX Laboratories received the samples on 10-12-22.

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,
- 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 TMW43102022 was designated 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. Sample TMW15102022 was designated to be spiked as MS/MSD on the chain of custody. Raw data for MS/MSD with each set of 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#22J151 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
TMW43102022	J151-01	10-11-22	S3VM	Anions by IC,
MW25102022	J151-02	10-11-22	S3VM	Anions by IC,
TMW31D102022	J151-03	10-11-22	S3VM	Anions by IC,
TMW15102022	J151-04	10-11-22	S3VM	Anions by IC,
MW35102022	J151-05	10-11-22	S3VM	Anions by IC,
MW35102022D	J151-06	10-11-22	S3VM	Anions by IC,
TMW13102022	J151-07	10-11-22	S3VM	Anions by IC,
TMW13102022D	J151-08	10-11-22	S3VM	Anions by IC,
TMW35102022	J151-09	10-11-22	S3VM	Anions by IC,
TMW34102022	J151-10	10-11-22	S3VM	Anions by IC,
TMW34102022D	J151-11	10-11-22	S3VM	Anions by IC,
TMW15102022MS	J151-04M	10-11-22	S3VM	Anions by IC,
TMW15102022MSD	J151-04S	10-11-22	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 except for Nitrate analysis for three samples*
		Analysis within 28 days for Bromide, Fluoride, Chloride and Sulfate	None. Holding times were met

*Initial analyses were all within 48-hour holding time. Dilutions for samples TMW15102022(J151-04), TMW13102022(J151-07) and TMW13102022D(J151-08) were analyzed few hours past holding time

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 1.2˚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:

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

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) =11/11X100=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 eleven 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 all initial analysis of water samples requested for this method. A total of eleven (11) water samples were collected on 10-11-22. Samples were initially analyzed on 10-12-22 and 10-13-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. However, dilutions for three samples (TMW15102022, TMW13102022 and TMW13102022D) were analyzed few hours past 48 hour holding time for Nitrate. Samples were re-analyzed on 10-31-22 and 11-01-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

4.12.2. 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 05-04-22 and 10-18-22. 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 05-04-22 and 10-18-22. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-12-22, 10-13-22, 10-31-22 and 11-01-22. A total of eleven 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 the 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.

4.12.3. Quality Control Samples consisted of two method blanks, two sets of LCS/LCSD, MS/MSD and sample/sample duplicate. Sample TMW15102022 was designated to be spiked as MS/MSD and sample/sample duplicate. One more set of MB and LCS/LCSD was analyzed with re-analysis of Chloride and Sulfate on 10-31-22. 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 and sample/sample duplicate for all anions.

Recoveries of MS/MSD were within acceptable limits except for few anions as indicated in the table below:

Anion	TMW17102022 MS%	TMW17102022 MSD%	% Acceptance limits
Bromide	52*	53*	91-110
Nitrate	77*	74*	88-111
Nitrite	75*	74*	87-111
O-Phosphate	62*	65*	80-116

*Outside Acceptance limits

4.12.4. Field duplicate sample and its associated sample: Sample MW35102022 was identified as field duplicate of MW35102022D, sample TMW13102022 was identified as field duplicate of TMW13102022D and sample TMW34102022 was identified as field duplicate of TMW34102022D. Results for each sample/sample duplicate is summarized in the tables below:

Anion	MW35102022 (Lab ID #J151-05) mg/L	MW35102022D (Lab ID #J151-06) mg/L	**% RPD**
Nitrate	U	U	--
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.58	0.58	<1
Chloride	320	300	6.45
Fluoride	0.20	0.19	5.13
Sulfate	1000	1000	<1

Anion	TMW13102022 (Lab ID #J151-07) mg/L	TMW13102022D (Lab ID #J151-08) mg/L	**% RPD**
Nitrate	2.8	2.8	<1
Nitrite	U	U	--
Orthophosphate	0.61	0.61	<1
Bromide	0.61	0.61	<1
Chloride	90	84	6.9
Fluoride	1.6	1.6	<1
Sulfate	360	340	5.71

Anion	TMW34102022 (Lab ID #J151-010) mg/L	TMW34102022D (Lab ID #J151-11) mg/L	**% RPD**
Nitrate	56	52	7.41
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.85	0.86	1.17
Chloride	220	270	20.4
Fluoride	0.24	0.25	4.08
Sulfate	1600	1700	6.06

4.12.5. Raw data was submitted for all requested field samples. Sample TMW43102022 (Lab ID#22J151-01) was designated 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. As mentioned in section 4.1.1., dilutions for three samples (TMW15102022, TMW13102022 and TMW13102022D) were analyzed about two hours past 48 hour holding time for Nitrate.

5.0 CONCLUSION

SDG #22J151 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.

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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J154 Analytical Data Package

Publication Date: 02-14-23

By
ZIBA HOSSEINI

14343 Peach Hill Rd.
Moorpark, CA, 93021

TABLE OF CONTENTS

EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2022 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 seventeen (17) water samples were collected on 10-10-22. EMAX Laboratories received the samples on 10-12-22. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample TMW53102022(Lab ID# J154-04) was designated as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation review. Sample TMW17102022 (Lab ID#J154-09) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Results and recoveries of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision. Raw data for method blank, LCS/LCSD and MS/MSD for each method 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, (both extraction and analysis), extraction logs, 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 methods of analysis:

EPA Method 3050B/8260C: Volatile Organics by GC-MS (17 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (15 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (15 samples)
EPA Method SW8332: Nitroglycerine and PETN (15 samples)
EPA Method 8081B: Organochlorine Pesticides (13 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (12 sample)

EPA Method 8151A: Chlorinated herbicides (12 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (13 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (12 samples)
EPA Method 6850: Perchlorate (14 samples)

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 Methods (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 extraction and analytical holding times were met for each method and all the related samples. The deviations, if any, are discussed in Section 4.0 for each method.

The analytical data evaluated in this data validation report (SDG # 22J154), has met the data quality and usability requirement as defined in the data quality objectives. While very few analytical QC exceedances were observed, it was not significant for any data qualifiers. 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 DoD *QSM 5.3, 2019.* The Approved site-specific Quality Assurance Project Plan (ECO QAPP) has the highest hierarchy.

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2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the 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 preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD),
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses 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. Next, selected analytical reports including QA/QC information was cross checked with raw data. The analysis and extraction sequence logs for each method were 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, surrogate recoveries, MS/MSD, and lab control samples (LCS/LCSD) 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 each analytical method requested on the chain of custody were submitted for all samples. One field sample TMW53102022 (Lab ID# J154-04) from this sample delivery group was designated as stage 3 data deliverable on the chain of custody. Raw data for this sample was evaluated comprehensively. Sample TMW17102022 (Lab ID#J154-09) was designated to be spiked as MS/MSD on the chain of custody for few methods only. Raw data for method blank, LCS/LCSD and MS/MSD was reviewed in detail. 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 regard to the requested method of analysis (collection to extraction and extraction 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
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated;
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards, and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

The following is a list of sample identifications and corresponding laboratory sample identification numbers:

Site Name: Fort Wingate, New Mexico

SDG#22J154 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW33102022	22J154-01	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW52102022	22J154-02	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
MW34102022	22J154-03	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW53102022	22J154-04	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW14A102022	22J154-05	10-10-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine &PETN Semivolatiles + APP9

Site Name: Fort Wingate, New Mexico

SDG#22J154 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
MW26102022	22J154-06	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
MW38102022	22J154-07	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW59102022	22J154-08	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW17102022	22J154-09	10-10-22	S3VM	VOCs by SW5030B/8260C, Perchlorate by 6850
TMW57102022	22J154-10	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides

Site Name: Fort Wingate, New Mexico

SDG#22J154 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
MW37102022	22J154-11	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
MW37102022D	22J154-12	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides Anions by IC
TMW61102022	22J154-13	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW03102022	22J154-14	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
FW31102022	22J154-15	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN
QC10102022 EB4	22J154-16	10-10-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides

Site Name: Fort Wingate, New Mexico

SDG#22J154 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
QC10102022 TB6	22J154-17	10-10-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW17102022MS	22J154-09M	10-10-22	S3VM	VOCs by SW5030B/8260C, Perchlorate by 6850
TMW17102022MSD	22J154-09S	10-10-22	S3VM	VOCs by SW5030B/8260C, Perchlorate by 6850

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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8330B	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in twenty ice preserved coolers.

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction, and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements 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"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	NA
Anions by IC	Water	Analysis 48 hours from collection for Nitrate, Nitrite &Orthophosphate and 28-days for Bromide, Chloride, Fluoride and Sulfate	NA
Mercury & Dissolved Mercury	Water	Collection to Analysis: 28 days	NA

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 and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in twenty ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 0.5˚C and as high as 4.3˚C upon arrival. All samples were received intact and 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 exactly the same way. 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. The completeness objective is to obtain a sufficient amount 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 5030B/8260C: VOCs) =17/17X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =15/15X100=100%
Completeness (EPA Method 8330B: Explosives) =15/15X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =15/15X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =13/13X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =12/12X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =12/12X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 13/13X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =12/12X100=100%
Completeness (EPA Method 6850: Perchlorate) =14/14X100=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 seventeen water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS

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. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Seventeen water samples were collected on 10-10-22. Samples were analyzed on 10-21-22, 10-22-22 and 10-24-22 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

Chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.1.7.

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to generation of initial calibration on

10-17-22 and at the beginning of each analysis shift on 10-21-22 and 10-24-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 10-17-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table:

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 10-17-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Minimum average response factors for all target compounds were within method’s recommended values, except for: Acetone (0.036) and 2-Butanone (0.014).

However, recoveries were within the requirement of 70-130%. Calibration check compounds (CCCs) met the acceptance criteria for %RSD among the response factors calculated for each level. Table 4.1.3.2 lists the CCCs with method requirement limits for %RSD among response factors for initial calibration.

Table 4.1.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Response Factors %RSD 10-17-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Initial calibration curve was verified by a second source standard on 10-17-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standard was analyzed at the beginning and end of each analysis shift on 10-21-22 and 10-24-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria. Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-21-22 (I&II)	Continuing cal. Response factors 10-24-22 (I&II)
		1st Analysis shift	2nd Analysis shift
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. The calculated % difference between RFs from continuing calibration and average response factors from initial calibration is summarized in Table 4.1.4.2 for continuing calibration reports presented with the data package.

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	**%Deviation From Initial calibration (Acceptance Limit)**	**%Deviation from Initial calibration (10-21-22)** I&II	**%Deviation from Initial calibration (10-24-22)** I&II
		1st Analysis shift	2nd Analysis shift
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list except for compounds in the table below for each daily standard:

Target Compounds	**%Deviation From In. calibration (Accept. Limit)**	**%Deviation from Initial calibration (10-21-22)** 1st Analysis shift		**%Deviation from Initial calibration (10-24-22)** 2nd Analysis shift	
		I	II	I	II
2-Chloroethylvinyl ether	≤ 20%	√	√	22.2*	22.2*
1,2-Dibromo-3chloropropane	≤ 20%	21.8*	√	√	22.8*
2-Chlorotoluene	≤ 20%	√	23.9*	√	√

*Outside control limits; I: Opening daily standard; II: Closing daily standard

These compounds were not detected in the corresponding field samples. This should not affect the data quality.

4.1.5. Quality Control samples reported consisted of two method blanks, two sets of LCS/LCSD and MS/MSD. Sample TMW17102022 (Lab ID#J154-09) was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for QC samples reported, were all within the project acceptance limits for the entire compound list.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blanks presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method's acceptable limits.

4.1.6. **Field duplicate sample** and its associated sample: Field sample MW37102022 was identified as field duplicate of sample MW37102022D. No Volatile Organic Compounds (VOCs) were detected in sample or field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample TMW53102022 (Lab ID# 22J154-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

4.2. SVOC (EPA Method 3520C/8270D)

4.2.1. Technical Holding Times

Holding time requirement was met for all fifteen samples. Water samples were collected on 10-10-22, extracted on 10-17-22 and were analyzed on 10-20-22 and 10-21-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of each analysis shift on 10-20-22 and 10-21-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group.

Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table.

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-07-22
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Average response factors for the rest of target compounds were within method's recommended values.

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 20%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-07-22
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 03-07-22
Benzoic acid	0.9969
2,4-Dinitrophenol	0.9964
4-Nitrophenol	0.9971
Di-n-Octyl phthalate	0.9972

All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of each analysis shift on 10-20-22 and 10-21-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (10-20-22) I&II	Continuing cal. Response factors (10-21-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√
2,4-Dinitrophenol	≥ 0.01	√	√
4-Nitrophenol	≥ 0.01	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Percent difference between RFs from continuing calibration and average response factors from initial calibration passed the method's criteria as summarized in Table 4.2.4.2.

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (10-20-22) I& II	Accepted Deviation from Initial calibration (10-21-22) I& II
Phenol	≤ 20	√	√
1,4-Dichlorobenzene	≤ 20	√	√
2-Nitrophenol	≤ 20	√	√
2,4-Dichlorophenol	≤ 20	√	√
Hexachlorobutadiene	≤ 20	√	√
4-Chloro-3-methylphenol	≤ 20	√	√
2,4,6-Trichlorophenol	≤ 20	√	√
Acenaphthene	≤ 20	√	√

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (10-20-22) I& II	Accepted Deviation from Initial calibration (10-21-22) I& II
N-Nitrosodiphenylamine	≤ 20	√	√
Pentachlorophenol	≤ 20	√	√
Fluoranthene	≤ 20	√	√
Di-n-Octylphthalate	≤ 20	√	√
Benzo(a)pyrene	≤ 20	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for some compounds in daily standard analyzed on 10-20-22, as listed in the table below:

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-20-22) 1st Analysis shift		%Deviation from Initial calibration (10-21-22) 2nd Analysis shift	
		I	II	I	II
1,4-Dinitrobenzene	≤ 20%	24.4*	21.3*	√	√
1,3-Dinitrobenzene	≤ 20%	22.2*	23.2*	√	√
2,4-Dinitrophenol	≤ 20%	35.9*	35.1*	√	√
2,6-Dinitrophenol	≤ 20%	--	22.0*	√	√
2,3,5-Trichlorophenol	≤ 20%	--	21.0*	√	√
2,4-Dinitrotoluene	≤ 20%	--	21.6*	√	√
4-Nitroaniline	≤ 20%	--	23.1*	√	√
4,6-Dinitro-2-methylphenol	≤ 20%	--	32.4*	√	√

*Outside control limits; √ denotes passing method acceptance limits

The exceedances were all high biased and the compounds were not detected in the corresponding field samples. This should not affect the data quality.

4.2.5. Quality Control samples reported consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries for the QC samples, LCS/LCSD reported were all within the project acceptance limits. Three compounds exceeded maximum 20% RPD limit in LCS/LCSD, (3/4 Methylphenol:25%), (Hexachloroethane:21%) and (Hexachloro-pentadiene:26%).

Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision in this method.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method’s acceptable limits, except surrogates listed in the table below:

Surrogate	MW38102022 (J154-07)	TMW03102022 (J154-14)	QC Limit%
2,4,6-Tribromophenol	31%*	√	43-140
2-Fluorophenol	15%*	√	19-119
Phenol-d5	27%*	√	40-130
Nitrobenzene	√	43%*	44-120

This was attributed to matrix interference in the method’s case narrative.

4.2.6. **Field duplicate sample** and its associated sample: Field sample MW37102022 was identified as field duplicate of sample MW37102022D. No Semi Volatile Organic Compounds (SVOCs) were detected in sample or field duplicate sample.

4.2.7. Raw data was submitted for all samples. Sample TMW53102022(Lab ID#J154-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.3. ORGANOCHLORINE PESTICIDES (EPA Method 3520C/ 8081B)

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen water samples requested for this method. Water samples were collected on 10-10-22, extracted on 10-16-22 and analyzed on 10-17-22 and 10-18-22 within required holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 09-02-22, 09-03-22 and before sample analysis on 10-17-22 and 10-18-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 09-02-22 and 09-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 08-11-22 for both channels. Percent RSD among the calibration factors was less than 15 for Toxaphene.

Retention time window width were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 08-11-22 and 09-12-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Performance of instrument was monitored by analysis of DDT and Endrin breakdown mixture. Before each continuing (daily) calibration a mixture of DDT and Endrin was analyzed. Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of four continuing calibration standards were analyzed at 10-injections interval on 10-17-22 and 10-18-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. In the four continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) and %RPDs were within established QC limits.

Results and recoveries of QC samples were confirmed with the reported raw data.

Results for method blank were reviewed for each component and no organochlorine pesticide was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Field sample MW37102022 was identified as field duplicate of sample MW37102022D. No Pesticides were detected in sample or field duplicate sample.

4.3.6. Raw data was submitted for sample and all QC samples. Sample TMW53102022 (Lab ID#22J154-04) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. Polychlorinated Biphenyls (EPA Method 3520C/ 8082A)

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Twelve water samples were collected on 10-10-22, extracted on 10-16-22, and analyzed on 10-19-22, within holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 09-07-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

Retention time window width were established at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 09-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-19-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Results and recoveries of LCS/LCSD was used for both accuracy and precision. Percent recoveries (%R) were within the established QC limits.

Results for method blank was reviewed for each component and no target analyte was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample and its associated sample: Field sample MW37102022 was identified as field duplicate of sample MW37102022D. No Polychlorinated Biphenyls (PCBs) were detected in sample or field duplicate sample.

4.4.6. Raw data was submitted for all samples. Sample TMW53102022 (Lab ID#J154-04) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Twelve water samples were collected on 10-10-22, extracted on 10-17-22 and analyzed on 10-19-22 and 10-20-22.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 10-03-22 and 10-04-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99819 was used for MCPP in column B.

Retention time windows were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 10-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-19-22, 10-20-22 and 10-21-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were all less than 20%.

Results for surrogate recoveries and QC were all reported from channel A. Channel B was used for confirmation only.

4.5.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. All herbicides target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits

Results for method blank was reviewed for each component and no Herbicide was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.5.5. Field duplicate sample and its associated sample: Field sample MW37102022 was identified as field duplicate of sample MW37102022D. No Chlorinated Herbicides were detected in sample or field duplicate sample.

4.5.6. Raw data was submitted for all samples. Sample TMW53102022 (Lab ID#J154-04) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Nitroaromatics by HPLC/UV (EPA Method 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for fifteen (15) water samples requested for this method. Water samples were collected on 10-10-22, extracted on 10-16-22 and analyzed on 10-17-22, 10-18-22 and 10-19-22, within holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis. Positive results, if any, were confirmed with UHPLC equipped with different column (Kinetex- Biphenyl column).

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-25-22 and 08-26-22. Confirmation (Kinetex-Biphenyl column) was calibrated on 08-23-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

Retention time windows were established for each target analyte at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for primary column on 08-26-22 and confirmation column on 08-23-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of seven continuing calibration standards were analyzed on 10-17-22, 10-18-22 and 10-19-22, bracketing the analyses of samples and all the QC samples. Five more daily standards were also analyzed with confirmation column on 10-17-22 and 10-19-22. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.6.5 Field duplicate sample and its associated sample**:** Field sample MW37102022 was identified as field duplicate of sample MW37102022D. No explosives were detected in the sample and associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample TMW53102022 (Lab ID#J154-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation column (Biphenyl column).

4.7. Nitroglycerine and PETN by UHPLC/UV (EPA Method 8332)

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for fifteen (15) water samples requested for this method. Water samples were collected on 10-10-22, extracted on 10-16-22 and analyzed on 10-18-22 and 10-19-22 within the holding time requirements.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis.

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on

08-24-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time windows were established for each target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 08-24-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of five continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-18-22 and 10-19-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Field sample MW37102022 was identified as field duplicate of sample MW37102022D. No explosive was detected in the sample and associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample TMW53102022 (Lab ID#J154-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.8. Total Petroleum hydrocarbons GRO (EPA Method 8015G)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen water samples requested for this method. Water samples were collected on 10-10-22. Samples were analyzed on 10-19-22 and 10-20-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Sample was carried through the system by purge and trap.

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-06-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10). Retention times for further sample analyses was used for peak identification and integration range.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-06-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-19-22 and 10-20-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW59102022 (Lab ID#J154-08) was selected to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.8.5. Field duplicate sample and its associated sample: Field sample MW37102022 was identified as field duplicate of sample MW37102022D. No TPH as Gasoline was detected in the sample and associated field duplicate sample.

4.8.6. Raw data was submitted for all samples. Sample TMW53102022 (Lab ID#J154-04) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve water sample requested for this method. Water samples were collected on 10-10-22, extracted on 10-15-22 and analyzed on 10-19-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Heavier range of total petroleum hydrocarbons were extracted and introduced into system by direct injection.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-08-22. Calibration factor (area for each compound/concentration) was used to quantify diesel range hydrocarbons (TPH as DRO). A second set of initial calibration curve was generated for lighter TPHs (Jet Fuel) and heavier TPHs (motor oil). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time window width was established by analysis of a window defining hydrocarbon standard (C10-C40). Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-08-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

A total of four continuing calibration standards were analyzed on 10-19-22 at 10-injections interval, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated for MS/MSD. Percent recoveries (%R) of LCS/LCSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.9.5. Field duplicate sample and its associated sample: Field sample MW37102022 was identified as field duplicate of sample MW37102022D. Traces of TPH as DRO was detected in the associated field duplicate sample as indicated in the table below:

TPH	MW37102022 (Lab ID #J154-11) mg/L	MW37102022D (Lab ID #J154-12) mg/L	**% RPD**
TPH as DRO	U	0.064J	200

4.9.6. Raw data was submitted for all samples. Sample TMW53102022 (Lab ID#J154-04) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.10. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for fourteen water samples requested for this method. Water samples were collected on 10-10-22. Samples were analyzed on 10-13-22, 10-14-22 and 10-17-22 within required holding time.

A High-Performance LC coupled with Mass Detector (HPLC/MS) was used for analysis.

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-21-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9960 (Perchlorate ion 83) and 0.9987 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

Retention time for each isotope (ion 83 and 85) at each calibration level was within 0.2 minutes required by the method.

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-21-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of eight daily standards were carried out on 10-13-22, 10-14-22 and 10-17-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of two method blanks, two sets of LCS/LCSD and MS/MSD. Sample TMW17102022 (Lab ID#J154-09) was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. No surrogate is used in this method.

4.10.5. Field duplicate sample and its associated sample: Field sample MW37102022 was identified as field duplicate of sample MW37102022D. No Perchlorate was detected in sample and associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample TMW53102022 (Lab ID#J154-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #22J154 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. 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.

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J157 Analytical Data Package

Publication Date: 02-28-23

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 2022 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 twelve (12) water samples were collected on 10-11-22. EMAX Laboratories received the samples on 10-12-22. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample TMW43102022(Lab ID# J157-01) was designated to be reviewed as stage 3 deliverable. No sample was designated for this purpose on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation review. Sample TMW15102022(Lab ID#J157-04) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Results and recoveries of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision. Raw data for method blank, LCS/LCSD and MS/MSD for each method 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, (both extraction and analysis), extraction logs, 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 methods of analysis:

EPA Method 3050B/8260C: Volatile Organics by GC-MS (12 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (7 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (6 samples)
EPA Method SW8332: Nitroglycerine and PETN (6 samples)
EPA Method 8081B: Organochlorine Pesticides (6 samples)

EPA Method 8082A: Polychlorinated Biphenyls; PCBs (3 sample)
EPA Method 8151A: Chlorinated herbicides (3 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (7 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (6 samples)
EPA Method 6850: Perchlorate (11 samples)

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 Methods (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 extraction and analytical holding times were met for each method and all the related samples. The deviations, if any, are discussed in Section 4.0 for each method.

The analytical data evaluated in this data validation report (SDG # 22J157), has met the data quality and usability requirement as defined in the data quality objectives. While very few analytical QC exceedances were observed, it was not significant for any data qualifiers. 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 DoD *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 twelve (12) water samples were collected on 10-11-22. EMAX Laboratories received the samples on 10-12-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the 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 preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if submitted
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses 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. Next, selected analytical reports including QA/QC information was cross checked with raw data. The analysis and extraction sequence logs for each method were 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, surrogate recoveries, MS/MSD, and lab control samples (LCS/LCSD) 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 each analytical method requested on the chain of custody were submitted for all samples. One field sample TMW43102022 (Lab ID# J157-01) from this sample delivery group was designated to be reviewed as stage 3 data deliverable. Raw data for this sample was evaluated comprehensively. Sample TMW15102022 (Lab ID#J157-04) was designated to be spiked as MS/MSD on the chain of custody for few methods only. Raw data for method blank, LCS/LCSD and MS/MSD was reviewed in detail. 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 regard to the requested method of analysis (collection to extraction and extraction 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
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated;
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards, and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

The following is a list of sample identifications and corresponding laboratory sample identification numbers:

Site Name: Fort Wingate, New Mexico

SDG#22J157 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW43102022	22J157-01	10-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
MW25102022	22J157-02	10-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW31D102022	22J157-03	10-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
TMW15102022	22J157-04	10-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
MW35102022	22J157-05	10-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides

Site Name: Fort Wingate, New Mexico

SDG#22J157 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
MW35102022D	22J157-06	10-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW13102022	22J157-07	10-11-22	S3VM	VOCs by SW5030B/8260C, Perchlorate by 6850
TMW13102022D	22J157-08	10-11-22	S3VM	VOCs by SW5030B/8260C, Perchlorate by 6850
TMW35102022	22J157-09	10-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
TMW34102022	22J157-10	10-11-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline; TPH as DRO Perchlorate by 6850
TMW34102022D	22J157-11	10-11-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline; TPH as DRO Perchlorate by 6850
QC11102022TB7	22J157-12	10-11-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW15102022MS	22J157-04M	10-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
TMW15102022MSD	22J157-04S	10-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850

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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8330B	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in twelve ice preserved coolers.

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction, and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements 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"
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	NA
Anions by IC	Water	Analysis 48 hours from collection for Nitrate, Nitrite &Orthophosphate and 28-days for Bromide, Chloride, Fluoride and Sulfate	NA
Mercury & Dissolved Mercury	Water	Collection to Analysis: 28 days	NA

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 and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in twelve ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler’s temperature was reported as low as 0.9˚C and as high as 5.2˚C upon arrival. All samples were received intact and 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 exactly the same way. 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. The completeness objective is to obtain a sufficient amount 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:

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

Completeness (EPA Method 5030B/8260C: VOCs) =12/12X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =7/7X100=100%
Completeness (EPA Method 8330B: Explosives) =6/6X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =6/6X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =6/6X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =3/3X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =3/3X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 7/7X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =6/6X100=100%
Completeness (EPA Method 6850: Perchlorate) =11/11X100=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 twelve water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS

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. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Twelve water samples were collected on 10-11-22. Samples were analyzed on 10-24-22 and 10-25-22 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

Chain-of-custody was reviewed for documentation of sample information and method of analysis. Qualification notations, if any, will be summarized in result section; section 4.1.7.

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to generation of initial calibration on 10-17-22 and at the beginning of analysis shift on 10-24-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 10-17-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table:

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 10-17-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.036) and 2-Butanone (0.014).

However, recoveries were within the requirement of 70-130%. Calibration check compounds (CCCs) met the acceptance criteria for %RSD among the response factors calculated for each level. Table 4.1.3.2 lists the CCCs with method requirement limits for %RSD among response factors for initial calibration.

Table 4.1.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Response Factors %RSD 10-17-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Initial calibration curve was verified by a second source standard on 10-17-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standard was analyzed at the beginning and end of analysis shift on 10-24-22 and 10-25-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-24-22	Continuing cal. Response factors 10-25-22
		Opening Daily St.	Closing Daily St.
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Calculated % difference between RFs from continuing calibration and average response factors from initial calibration is summarized in Table 4.1.4.2 for continuing calibration reports presented with the data package.

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	**%Deviation From Initial calibration (Acceptance Limit)**	**%Deviation from Initial calibration (10-24-22)**	**%Deviation from Initial calibration (10-25-22)**
		Opening Daily St.	Closing Daily St
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list except for compounds in the table below for each daily standard:

Target Compounds	**%Deviation From In. calibration (Accept. Limit)**	**%Deviation from Initial calibration (10-24-22)**	**%Deviation from Initial calibration (10-25-22)**
		Opening Daily St.	Closing Daily St
Bromomethane	≤ 20%	√	23.8*
2-Butanone	≤ 20%	√	21.4*
2-Chloroethylvinyl ether	≤ 20%	√	40.7*
1,2-Dibromo-3-chloropropane	≤ 20%	√	27.7*
1,2,4-Trichlorobenzene	≤ 20%	√	27.0*
Naphthalene	≤ 20%	√	24.3*
1,23-Trichlorobenzene	≤ 20%	√	27.0*

*Outside control limits;

These compounds were not detected in the corresponding field samples. This should not affect the data quality.

4.1.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW15102022 (Lab ID#J157-04) was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for QC samples reported, were all within the project acceptance limits for the entire compound list.

Results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blanks presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method's acceptable limits.

4.1.6. **Field duplicate sample** and its associated sample: Field samples MW35102022 was identified as field duplicate of sample MW35102022D, sample TMW13102022 was identified as field duplicate of TMW13102022D and sample TMW34102022 was identified as field duplicate of TMW34102022D. No Volatile Organic Compounds (VOCs) were detected in each sample or field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample TMW43102022 (Lab ID# 22J157-01) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

4.2. SVOC (EPA Method 3520C/8270D)

4.2.1. Technical Holding Times

Holding time requirement was met for all seven samples. Water samples were collected on 10-11-22, extracted on 10-18-22 and were analyzed on 10-27-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 10-27-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group.

Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table.

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-07-22
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Average response factors for the rest of target compounds were within method's recommended values.

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 20%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-07-22
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 03-07-22
Benzoic acid	0.9969
2,4-Dinitrophenol	0.9964
4-Nitrophenol	0.9971
Di-n-Octyl phthalate	0.9972

All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-27-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (10-27-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Percent difference between RFs from continuing calibration and average response factors from initial calibration passed the method's criteria as summarized in Table 4.2.4.2.

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (10-27-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (10-27-22) I& II
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for some compounds in daily standard analyzed on 10-20-22, as listed in the table below:

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-27-22)	
		Opening Daily St.	Closing Daily St.
Pentachlorophenol	≤ 20%	√	22.9*

*Outside QC limit √ Denotes passing method acceptance limits

The compound was not detected in the corresponding field samples. This should not affect the data quality.

4.2.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW15102022 (Lab ID#J157-04) was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries for the LCS/LCSD reported were all within the project acceptance limits. Recoveries of MS/MSD were all within acceptance limits except for one compound listed in the table below:

Compound	BGMW11102022 (J236-04) MS%	BGMW11102022 (J236-04) MSD%	QC Limit%
Phenol	65	48*	50-130

*Outside QC limit

Therefore, the recoveries of MS and LCS/LCSD were used to evaluate the QC. Quite a number of compounds failed maximum 20%RPD limits in MS/MSD. These compounds were not detected in any of the field samples. Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision for these compounds

Results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method's acceptable limits.

4.2.6. **Field duplicate sample** and its associated sample: Field sample MW35102022 was identified as field duplicate of sample MW35102022D. No Semi Volatile Organic Compounds (SVOCs) were detected in sample or field duplicate sample.

4.2.7. Raw data was submitted for all samples. Sample TMW43102022(Lab ID#J157-01) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.3. ORGANOCHLORINE PESTICIDES (EPA Method 3520C/ 8081B)

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six water samples requested for this method. Water samples were collected on 10-11-22, extracted on 10-18-22 and analyzed on 10-19-22 and 10-20-22 within required holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 09-02-22, 09-03-22 and before sample analysis on 10-19-22 and 10-20-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 09-02-22 and 09-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 08-11-22 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene.

Retention time window width were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 08-11-22 and 09-12-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Performance of instrument was monitored by analysis of DDT and Endrin breakdown mixture. Before each continuing (daily) calibration a mixture of DDT and Endrin was analyzed. Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of four continuing calibration standards were analyzed at 10-injections interval on 10-19-22 and 10-20-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. However, few compounds exceeded maximum percent difference in the opening daily standard analyzed on 10-20-22 (channel A), as summarized in the table below:

Target Compounds	**%Deviation From Initial calibration (Acceptance Limit)**	**%Deviation from Initial calibration (10-20-22) Opening Daily St.**
		Channel A
DDT	≤ 20%	34*
Methoxychlor	≤ 20%	35*
Endrin Ketone	≤ 20%	28*

*Outside QC limit

The exceedances were all high biased and none of these compounds were detected in the field samples. In the four continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) and %RPDs were within established QC limits.

Results and recoveries of QC samples were confirmed with the reported raw data.

Results for method blank were reviewed for each component and no organochlorine pesticide was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Field sample MW35102022 was identified as field duplicate of sample MW35102022D. No Pesticides were detected in sample or field duplicate sample.

4.3.6. Raw data was submitted for sample and all QC samples. Sample TMW43102022 (Lab ID#22J157-01) was designated as stage 3 data deliverable on the chain of custody. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. Polychlorinated Biphenyls (EPA Method 3520C/ 8082A)

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Three water samples were collected on 10-11-22, extracted on 10-18-22, and analyzed on 10-21-22, within holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 09-07-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

Retention time window width were established at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 09-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-21-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Results and recoveries of LCS/LCSD was used for both accuracy and precision. Percent recoveries (%R) were within the established QC limits.

Results for method blank was reviewed for each component and no target analyte was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample and its associated sample: Field sample MW35102022 was identified as field duplicate of sample MW35102022D. No Polychlorinated Biphenyls (PCBs) were detected in sample or field duplicate sample.

4.4.6. Raw data was submitted for all samples. Sample MW25102022(Lab ID#J157-02) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Three water samples were collected on 10-11-22, extracted on 10-14-22 and analyzed on 10-17-22 and 10-18-22.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 10-03-22 and 10-04-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99819 was used for MCPP in column B.

Retention time windows were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 10-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-17-22 and 10-18-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were all less than 20%, except for the compounds summarized in the table below in the closing daily standard:

Target Compounds	**%Deviation From Initial calibration (Accept. Limit)**	**%Deviation from Initial calibration (10-18-22) Opening daily std.**		**%Deviation from Initial calibration (10-18-22) Closing daily std.**	
		Column A	Column B	Column A	Column B
Acifluorfen	≤ 20%	23*	33*	28*	35*
Dinoseb	≤ 20%	√	26*	√	29*

*Outside QC limit

None of these compounds were detected in the field samples, and it should not affect the quality of data.

Results for surrogate recoveries and QC were all reported from channel A. Channel B was used for confirmation only.

4.5.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. All herbicides target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits

Results for method blank was reviewed for each component and no Herbicide was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.5.5. Field duplicate sample and its associated sample: Field sample MW35102022 was identified as field duplicate of sample MW35102022D. No Chlorinated Herbicides were detected in sample or field duplicate sample.

4.5.6. Raw data was submitted for all samples. Sample MW25102022(Lab ID#J157-02) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Nitroaromatics by HPLC/UV (EPA Method 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six (6) water samples requested for this method. Water samples were collected on 10-11-22, extracted on 10-13-22 and analyzed on 10-14-22 and 10-15-22, within holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis. Positive results, if any, were confirmed with UHPLC equipped with different column (Kinetex- Biphenyl column).

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-25-22 and 08-26-22. Confirmation (Kinetex-Biphenyl column) was calibrated on 08-23-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

Retention time windows were established for each target analyte at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for primary column on 08-26-22 and confirmation column on 08-23-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of four continuing calibration standards were analyzed on 10-14-22 and 10-15-22, bracketing the analyses of samples and all the QC samples. Three more daily standards were also analyzed with confirmation column on 10-17-22. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW15102022 (Lab ID#J157-04) was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.6.5 Field duplicate sample and its associated sample**:** Field sample MW35102022 was identified as field duplicate of sample MW35102022D. No explosives were detected in the sample and associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample TMW43102022(Lab ID#J157-01) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation column (Biphenyl column).

4.7. Nitroglycerine and PETN by UHPLC/UV (EPA Method 8332)

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six (6) water samples requested for this method. Water samples were collected on 10-11-22, extracted on 10-13-22 and analyzed on 10-14-22 within the holding time requirements.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis.

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 08-24-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time windows were established for each target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 08-24-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-14-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW15102022 (Lab ID#J157-04) was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Field sample MW35102022 was identified as field duplicate of sample MW35102022D. No explosive was detected in the sample and associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample TMW43102022(Lab ID#J157-01) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.8. Total Petroleum hydrocarbons GRO (EPA Method 8015G)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven water samples requested for this method. Water samples were collected on 10-11-22. Samples were analyzed on 10-18-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Sample was carried through the system by purge and trap.

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 08-04-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10). Retention times for further sample analyses was used for peak identification and integration range.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 08-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-18-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was assigned to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.8.5. Field duplicate sample and its associated sample: Field sample MW35102022 was identified as field duplicate of sample MW35102022D and field sample TMW34102022 was identified as field duplicate of sample TMW34102022D. No TPH as Gasoline was detected in each sample and associated field duplicate sample.

4.8.6. Raw data was submitted for all samples. Sample MW25102022(Lab ID#J157-02) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six water sample requested for this method. Water samples were collected on 10-11-22, extracted on 10-15-22 and analyzed on 10-18-22 and 10-19-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Heavier range of total petroleum hydrocarbons were extracted and introduced into system by direct injection.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-08-22. Calibration factor (area for each compound/concentration) was used to quantify diesel range hydrocarbons (TPH as DRO). A second set of initial calibration curve was generated for lighter TPHs (Jet Fuel) and heavier TPHs (motor oil). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time window width was established by analysis of a window defining hydrocarbon standard (C10-C40). Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-08-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

A total of three continuing calibration standards were analyzed on 10-18-22 and 10-19-22 at 10-injections interval, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) of LCS/LCSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.9.5. Field duplicate sample and its associated sample: Field sample MW35102022 was identified as field duplicate of sample MW35102022D and Field sample TMW34102022 was identified as field duplicate of sample TMW34102022D. Traces of TPH as DRO was detected in sample and associated field duplicate sample as indicated in the table below:

TPH	MW35102022 (Lab ID #J157-05) mg/L	MW35102022D (Lab ID #J157-06) mg/L	**% RPD**
TPH as DRO	U	U	--

TPH	TMW34102022 (Lab ID #J157-10) mg/L	TMW34102022D (Lab ID #J157-11) mg/L	**% RPD**
TPH as DRO	0.11J	0.071J	43.1

4.9.6. Raw data was submitted for all samples. Sample MW25102022(Lab ID#J157-02) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.10. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eleven water samples requested for this method. Water samples were collected on 10-11-22. Samples were analyzed on 10-14-22, 10-15-22 and 10-17-22 within required holding time.

A High-Performance LC coupled with Mass Detector (HPLC/MS) was used for analysis.

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-21-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9960 (Perchlorate ion 83) and 0.9987 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

Retention time for each isotope (ion 83 and 85) at each calibration level was within 0.2 minutes required by the method.

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-21-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of seven daily standards were carried out on 10-14-22, 10-15-22 and 10-17-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of two method blanks, two sets of LCS/LCSD and MS/MSD. Sample TMW15102022 (Lab ID#J157-04) was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. No surrogate is used in this method.

4.10.5. Field duplicate sample and its associated sample: Field sample MW35102022 was identified as field duplicate of sample MW35102022D, field sample TMW13102022 was identified as field duplicate of sample TMW13102022D and field sample TMW34102022 was identified as field duplicate of sample TMW34102022D. No Perchlorate was detected in each sample and associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample TMW43102022(Lab ID#J157-01) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #22J157 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. 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.

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J171 Analytical Data Package

Publication Date: 02-28-2023

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 2022 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 eleven (11) water samples were collected on 10-12-22. EMAX Laboratories received the samples on 10-13-22. 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 MW20102022 (Lab ID#J171-02) was designated 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 analysis on the chain of custody. Recoveries and results of LCS/LCSD was used to evaluate accuracy and precision. Raw data for method blanks and LCS/LCSDs were also 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 (11 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 # 22J171) 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 eleven (11) water samples were collected on 10-12-22. EMAX Laboratories received the samples on 10-13-22.

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, if requested;
- 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 MW20102022 was designated on the chain of custody 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 designated to be spiked as MS/MSD on the chain of custody. Raw data each set of LCS/LCSD was also 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#22J171 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
BGMW09102022	J171-01	10-12-22	S3VM	Anions by IC,
MW20102022	J171-02	10-12-22	S3VM	Anions by IC,
MW18D102022	J171-03	10-12-22	S3VM	Anions by IC,
TMW25102022	J171-04	10-12-22	S3VM	Anions by IC,
BGMW13S102022	J171-05	10-12-22	S3VM	Anions by IC,
BGMW13S102022D	J171-06	10-12-22	S3VM	Anions by IC,
TMW06102022	J171-07	10-12-22	S3VM	Anions by IC,
TMW38102022	J171-08	10-12-22	S3VM	Anions by IC,
BGMW13D102022	J171-09	10-12-22	S3VM	Anions by IC,
TMW45102022	J171-10	10-12-22	S3VM	Anions by IC,
QC12102022EB5	J171-11	10-12-22	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
		Analysis within 28 days for Bromide, Fluoride, Chloride and Sulfate	None. Holding times were met

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:

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

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. The completeness objective is to obtain enough valid data to enable the goals and objectives of the project to be achieved. One sample (BGMW03102022) was not analyzed due to being mistakenly preserved. Only the corresponding field duplicate (BGMW03102022D) was analyzed.

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:

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

Completeness (EPA Method 9056A: Anions) =11/11X100=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 eleven 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 eleven (11) water samples were collected on 10-12-22. Samples were analyzed on 10-13-22 and 10-14-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Some samples were re-analyzed with dilution on 10-28-22, 10-29-22 and 10-31-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

4.12.2. 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 05-04-22 and 10-18-22. 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 05-04-22 and 10-18-22. Recoveries were all within

90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-13-22, 10-14-22, 10-28-22, 10-29-22 and 10-31-22. A total of fourteen 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 the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.12.3. Quality Control Samples consisted of two method blanks and two sets of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Two more sets of LCS/LCSD were analyzed with Chloride and Sulfate analysis on 10-28-22 and 10-31-22. Recoveries of LCS/LCSDs were all within 90-110 % of spiked values for each anion.

Percent RPDs were less than 20% for each set of LCS/LCSD for all anions.

4.12.4. Field duplicate sample and its associated sample: Sample BGMW13S102022 was identified as field duplicate of BGMW13S102022D. Results for sample/sample duplicate is summarized in the table below:

Anion	BGMW13S102022 (Lab ID #J171-05) mg/L	BGMW13S102022D (Lab ID #J171-06) mg/L	**% RPD**
Nitrate	U	U	--
Nitrite	U	U	--
Orthophosphate	0.51	0.52	1.94
Bromide	0.45J	0.45J	<1
Chloride	110	150	30.8
Fluoride	1.0	1.1	9.52
Sulfate	42	39	7.41

4.12.5. Raw data was submitted for all requested field samples. Sample MW20102022 (Lab ID#22J171-02) was designated 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 #22J171 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.

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

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

Project No: Eco-18-1237

SDG #22J174 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 2022 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 twelve (12) water samples were collected on 10-12-22. EMAX Laboratories received the samples on 10-13-22. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample MW20102022(Lab ID# J174-02) was designated to be reviewed as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation review. No sample was designated to be spiked as MS/MSD on the chain of custody. Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision. Raw data for method blank and LCS/LCSD for each method 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, (both extraction and analysis), extraction logs, 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 methods of analysis:

EPA Method 3050B/8260C: Volatile Organics by GC-MS (12 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (9 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (11 samples)
EPA Method SW8332: Nitroglycerine and PETN (11 samples)
EPA Method 8081B: Organochlorine Pesticides (8samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (5 sample)
EPA Method 8151A: Chlorinated herbicides (5 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (8 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (7 samples)
EPA Method 6850: Perchlorate (9 samples)

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 Methods (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 extraction and analytical holding times were met for each method and all the related samples. The deviations, if any, are discussed in Section 4.0 for each method.

The analytical data evaluated in this data validation report (SDG # 22J174) has met the data quality and usability requirement as defined in the data quality objectives. While very few analytical QC exceedances were observed, it was not significant for any data qualifiers. 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 DoD *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 twelve (12) water samples were collected on 10-12-22. EMAX Laboratories received the samples on 10-13-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the 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 preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if submitted
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses 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. Next, selected analytical reports including QA/QC information was cross checked with raw data. The analysis and extraction sequence logs for each method were 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, surrogate recoveries, MS/MSD, and lab control samples (LCS/LCSD) 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 each analytical method requested on the chain of custody were submitted for all samples. One field sample MW20102022 (Lab ID# J174-02) from this sample delivery group was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample was evaluated comprehensively. No sample was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank and LCS/LCSD was reviewed in detail. 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 regard to the requested method of analysis (collection to extraction and extraction 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
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated; if applicable
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards, and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

The following is a list of sample identifications and corresponding laboratory sample identification numbers:

Site Name: Fort Wingate, New Mexico

SDG#22J174 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
BGMW09102022	22J174-01	10-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Polychlorinated Biphenyls Chlorinated Herbicides Perchlorate by 6850
MW20102022	22J174-02	10-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO
MW18D102022	22J174-03	10-12-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline; TPH as DRO Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
TMW25102022	22J174-04	10-12-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine &PETN
BGMW13S102022	22J174-05	10-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW06102022	22J174-06	10-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine &PETN TPH Gasoline; TPH as DRO

Site Name: Fort Wingate, New Mexico

SDG#22J174 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
BGMW13S102022D	22J174-07	10-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW38102022	22J174-08	10-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
BGMW13D102022	22J174-09	10-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW45102022	22J174-10	10-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
QC12102022EB5	22J174-11	10-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
QC12102022TB8	22J174-12	10-11-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8330B	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in thirteen ice preserved coolers.

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction, and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements 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"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	NA
Anions by IC	Water	Analysis 48 hours from collection for Nitrate, Nitrite &Orthophosphate and 28-days for Bromide, Chloride, Fluoride and Sulfate	NA
Mercury & Dissolved Mercury	Water	Collection to Analysis: 28 days	NA

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 and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in thirteen ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.8˚C and as high as 4.8˚C upon arrival. All samples were received intact and 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 exactly the same way. 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:

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

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. The completeness objective is to obtain a sufficient amount 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:

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

Completeness (EPA Method 5030B/8260C: VOCs) =12/12X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =9/9X100=100%
Completeness (EPA Method 8330B: Explosives) =11/11X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =11/11X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =8/8X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =5/5X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =5/5X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 8/8X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =7/7X100=100%
Completeness (EPA Method 6850: Perchlorate) =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 twelve water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS

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. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Twelve water samples were collected on 10-12-22. Samples were analyzed on 10-24-22, 10-25-22and 10-26-22 within method's requirement for holding time. (Water samples were preserved with hydrochloric acid).

Chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.1.7.

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to generation of initial calibration on

10-17-22 and at the beginning of each analysis shift on 10-24-22, 10-25-22 and 10-26-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 10-17-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table:

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 10-17-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.036) and 2-Butanone (0.014).

However, recoveries were within the requirement of 70-130%. Calibration check compounds (CCCs) met the acceptance criteria for %RSD among the response factors calculated for each level. Table 4.1.3.2 lists the CCCs with method requirement limits for %RSD among response factors for initial calibration.

Table 4.1.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Response Factors %RSD 10-17-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Initial calibration curve was verified by a second source standard on 10-17-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standard was analyzed at the beginning and end of each analysis shift on 10-24-22, 10-25-22 and 10-26-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-24-22	Continuing cal. Response factors 10-25-22	Continuing cal. Response factors 10-26-22
		Opening & closing Daily St.	Opening &closing Daily St.	Opening &closing Daily St.
Chloromethane	≥ 0.10	√	√	√
1,1-Dichloroethane	≥ 0.20	√	√	√
Chlorobenzene	≥ 0.50	√	√	√
Bromoform	≥ 0.10	√	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. The calculated % difference between RFs from continuing calibration and average response factors from initial calibration is summarized in Table 4.1.4.2 for continuing calibration reports presented with the data package.

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-24-22)	%Deviation from Initial calibration (10-25-22)	%Deviation from Initial calibration (10-26-22)
		Opening & closing Daily St.	Opening & closing Daily St.	Opening & closing Daily St.
Vinyl chloride	≤ 20%	√	√	√
1,1-Dichloroethene	≤ 20%	√	√	√
Chloroform	≤ 20%	√	√	√
1,2-Dichloropropane	≤ 20%	√	√	√
Toluene	≤ 20%	√	√	√
Ethyl benzene	≤ 20%	√	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list except for compounds in the table below for closing daily standard in the first analysis shift:

Target Compounds	%Deviation From In. calibration (Accept. Limit)	%Deviation from Initial calibration (10-24-22)	%Deviation from Initial calibration (10-25-22)
		Opening Daily St.	Closing Daily St
Bromomethane	≤ 20%	√	23.8*
2-Butanone	≤ 20%	√	21.4*
2-Chloroethylvinyl ether	≤ 20%	√	40.7*
1,2-Dibromo-3-chloropropane	≤ 20%	√	27.7*
1,2,4-Trichlorobenzene	≤ 20%	√	27.0*
Naphthalene	≤ 20%	√	24.3*
1,23-Trichlorobenzene	≤ 20%	√	27.0*

*Outside control limits;

These compounds were not detected in the corresponding field samples. This should not affect the data quality.

4.1.5. Quality Control samples reported consisted of three method blanks, and three sets of LCS/LCSD. No sample was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were all within the project acceptance limits for the entire compound list. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blanks presented with the data package, analyzed with samples did not show presence of any target compounds.
Surrogate recoveries were all within the method's acceptable limits.

4.1.6. **Field duplicate sample** and its associated sample: Field sample BGMW13S102022 was identified as field duplicate of sample BGMW13S102022D. No Volatile Organic Compounds (VOCs) were detected in sample or field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample MW20102022 (Lab ID# 22J174-02) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

4.2. SVOC (EPA Method 3520C/8270D)

4.2.1. Technical Holding Times

Holding time requirement was met for all nine samples. Water samples were collected on 10-12-22, extracted on 10-19-22 and were analyzed on 11-02-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 11-02-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group.

Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table.

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-07-22
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Average response factors for the rest of target compounds were within method's recommended values.

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 20%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-07-22
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 03-07-22
Benzoic acid	0.9969
2,4-Dinitrophenol	0.9964
4-Nitrophenol	0.9971
Di-n-Octyl phthalate	0.9972

All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 11-02-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (11-02-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Percent difference between RFs from continuing calibration and average response factors from initial calibration passed the method's criteria as summarized in Table 4.2.4.2.

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (11-02-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√

Calibration Check Compounds (CCCs)	**% Deviation From Initial calibration (Acceptance Limit)**	**Accepted Deviation from Initial calibration (11-02-22) I& II**
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for some compounds in closing daily standard, as listed in the table below:

Target Compounds	**%Deviation From Initial calibration (Acceptance Limit)**	**%Deviation from Initial calibration (11-02-22)**	
		Opening Daily St.	Closing Daily St.
2,4-Dinitrophenol	≤ 20%	23.7*	√
Phenol	≤ 20%	√	20.7*
2-Methylphenol	≤ 20%	√	23.1*
Bis(2-Chloroisopropyl) ether	≤ 20%	√	27.7*
4-Methylphenol	≤ 20%	√	20.9*
4-Chloro-3-methylphenol	≤ 20%	√	24.8*
Fluoranthene	≤ 20%	√	25.6*
Bis(2-Ethylhexyl) phthalate	≤ 20%	√	21.2*

*Outside QC limit √ Denotes passing method acceptance limits

These compounds were not detected in the corresponding field samples. This should not affect the data quality.

4.2.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries for the LCS/LCSD reported were all within the project acceptance limits.

Recoveries of LCS/LCSD were used to evaluate both accuracy and precision. Three compounds failed maximum 20%RPD limits in LCS/LCSD; Caprolactam:34%, Benzaldehyde:46% and 1,2,4,5-Tetrachlorobenzene:55%. These compounds were not detected in any of the field samples.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.
Surrogate recoveries were all within the method's acceptable limits.

4.2.6. **Field duplicate sample** and its associated sample: Field sample BGMW13S102022 was identified as field duplicate of sample BGMW13S102022D. No Semi Volatile Organic Compounds (SVOCs) were detected in sample or field duplicate sample.

4.2.7. Raw data was submitted for all samples. Sample MW20102022(Lab ID#J174-02) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.3. ORGANOCHLORINE PESTICIDES (EPA Method 3520C/ 8081B)

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight water samples requested for this method. Water samples were collected on 10-12-22, extracted on 10-17-22 and analyzed on 10-18-22 within required holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 09-02-22, 09-03-22 and before sample analysis on 10-18-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 09-02-22 and 09-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 08-11-22 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene.

Retention time window width were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 08-11-22 and 09-12-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Performance of instrument was monitored by analysis of DDT and Endrin breakdown mixture. Before each continuing (daily) calibration a mixture of DDT and Endrin was analyzed. Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval on 10-18-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. In the three continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) and %RPDs were within established QC limits.

Results and recoveries of QC samples were confirmed with the reported raw data.

Results for method blank were reviewed for each component and no organochlorine pesticide was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Field sample BGMW13S102022 was identified as field duplicate of sample BGMW13S102022D. No Pesticides were detected in sample or field duplicate sample.

4.3.6. Raw data was submitted for sample and all QC samples. Sample MW20102022 (Lab ID#22J174-02) was designated as stage 3 data deliverable on the chain of custody. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. Polychlorinated Biphenyls (EPA Method 3520C/ 8082A)

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Five water samples were collected on 10-12-22, extracted on 10-17-22, and analyzed on 10-19-22, within holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 09-07-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

Retention time window width were established at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 09-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-19-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Results and recoveries of LCS/LCSD was used for both accuracy and precision. Percent recoveries (%R) were within the established QC limits.

Results for method blank was reviewed for each component and no target analyte was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample and its associated sample: Field sample BGMW13S102022 was identified as field duplicate of sample BGMW13S102022D. No Polychlorinated Biphenyls (PCBs) were detected in sample or field duplicate sample.

4.4.6. Raw data was submitted for all samples. Sample BGMW09102022(Lab ID#J174-01) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Five water samples were collected on 10-12-22, extracted on 10-19-22 and analyzed on 10-21-22 and 10-22-22.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 10-03-22 and 10-04-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99819 was used for MCPP in column B.

Retention time windows were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 10-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-21-22 and 10-22-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were all less than 20%, except for the compounds summarized in the table below in the closing daily standard:

Target Compounds	%Deviation From Initial calibration (Accept. Limit)	%Deviation from Initial calibration (10-21-22) Opening daily std.		%Deviation from Initial calibration (10-22-22) Closing daily std.	
		Column A	Column B	Column A	Column B
MCPP	≤ 20%	√	25*	√	24*
Acifluorfen	≤ 20%	√	√	√	26*

*Outside QC limit

None of these compounds were detected in the field samples, and it should not affect the quality of data.

Results for surrogate recoveries and QC were all reported from channel A. Channel B was used for confirmation only.

4.5.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. All herbicides target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits

Results for method blank was reviewed for each component and no Herbicide was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.5.5. Field duplicate sample and its associated sample: Field sample BGMW13S102022 was identified as field duplicate of sample BGMW13S102022D. No Chlorinated Herbicides were detected in sample or field duplicate sample.

4.5.6. Raw data was submitted for all samples. Sample BGMW09102022(Lab ID#J174-01) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Nitroaromatics by HPLC/UV (EPA Method 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eleven (11) water samples requested for this method. Water samples were collected on 10-12-22, extracted on 10-17-22 and analyzed on 10-18-22 and 10-19-22, within holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis. Positive results, if any, were confirmed with UHPLC equipped with different column (Kinetex- Biphenyl column).

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-25-22 and 08-26-22. Confirmation (Kinetex-Biphenyl column) was calibrated on 08-23-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

Retention time windows were established for each target analyte at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for primary column on 08-26-22 and confirmation column on 08-23-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of four continuing calibration standards were analyzed on 10-18-22 and 10-19-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.6.5 Field duplicate sample and its associated sample**:** Field sample BGMW13S102022 was identified as field duplicate of sample BGMW13D102022D. No explosives were detected in the sample and associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample MW20102022(Lab ID#J174-02) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.7. Nitroglycerine and PETN by UHPLC/UV (EPA Method 8332)

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eleven (11) water samples requested for this method. Water samples were collected on 10-12-22, extracted on 10-17-22 and analyzed on 10-18-22 within the holding time requirements.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis.

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 08-24-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time windows were established for each target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 08-24-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-18-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Field sample BGMW13S102022 was identified as field duplicate of sample BGMW13S102022D. No explosive was detected in the sample and associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample MW20102022(Lab ID#J174-02) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.8. Total Petroleum hydrocarbons GRO (EPA Method 8015G)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight water samples requested for this method. Water samples were collected on

10-12-22. Samples were analyzed on 10-21-22, 10-22-22 and 10-25-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Sample was carried through the system by purge and trap.

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 08-04-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10). Retention times for further sample analyses was used for peak identification and integration range.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 08-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Five continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-21-22, 10-22-22 and 10-25-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of two method blanks and two sets of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.8.5. Field duplicate sample and its associated sample: Field sample BGMW13S102022 was identified as field duplicate of sample BGMW13S102022D. No TPH as Gasoline was detected in sample and associated field duplicate sample.

4.8.6. Raw data was submitted for all samples. Sample MW20102022(Lab ID#J174-02) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven water sample requested for this method. Water samples were collected on 10-12-22, extracted on 10-15-22 and analyzed on 10-19-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Heavier range of total petroleum hydrocarbons were extracted and introduced into system by direct injection.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-08-22. Calibration factor (area for each compound/concentration) was used to quantify diesel range hydrocarbons (TPH as DRO). A second set of initial calibration curve was generated for lighter TPHs (Jet Fuel) and heavier TPHs (motor oil). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time window width was established by analysis of a window defining hydrocarbon standard (C10-C40). Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-08-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

A total of four continuing calibration standards were analyzed on 10-19-22 at 10-injections interval, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) of LCS/LCSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.9.5. Field duplicate sample and its associated sample: Field sample BGMW13S102022 was identified as field duplicate of sample BGMW13S102022D. No TPH as DRO was detected in sample and associated field duplicate sample.

4.9.6. Raw data was submitted for all samples. Sample MW20102022(Lab ID#J174-02) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.10. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eleven water samples requested for this method. Water samples were collected on 10-12-22. Samples were analyzed on 10-17-22 and 10-18-22 within required holding time.

A High-Performance LC coupled with Mass Detector (HPLC/MS) was used for analysis.

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-21-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9960 (Perchlorate ion 83) and 0.9987 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

Retention time for each isotope (ion 83 and 85) at each calibration level was within 0.2 minutes required by the method.

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-21-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of five daily standards were carried out on 10-17-22 and 10-18-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of one method blank, set of LCS/LCSD and MS/MSD. Sample TMW38102022 (Lab ID#J174-08) was selected to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. No surrogate is used in this method.

4.10.5. Field duplicate sample and its associated sample: Field sample BGMW13S102022 was identified as field duplicate of sample BGMW13S102022D. No Perchlorate was detected in sample and associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample MW20102022(Lab ID#J174-02) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #22J174 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. 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.

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J213 Analytical Data Package

Publication Date: 02-28-2023

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 2022 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 eight (8) water samples were collected on 10-13-22. EMAX Laboratories received the samples on 10-14-22. 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 MW22D102022 (Lab ID#J213-03) was designated 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. Sample BGMW11102022(Lab ID#J213-04) was designated to be spiked as MS/MSD on the chain of custody. Recoveries and results of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision. Raw data for method blanks, MS/MSD and LCS/LCSDs were also 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 (8 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 # 22J213) 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 eight (8) water samples were collected on 10-13-22. EMAX Laboratories received the samples on 10-14-22.

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,
- 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 MW22102022 was designated 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. Sample BGMW11102022 was designated to be spiked as MS/MSD on the chain of custody. Raw data for MS/MSD with each set of 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#22J213 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
MW39102022	J213-01	10-13-22	S3VM	Anions by IC,
TMW32102022	J213-02	10-13-22	S3VM	Anions by IC,
MW22D102022	J213-03	10-13-22	S3VM	Anions by IC,
BGMW11102022	J213-04	10-13-22	S3VM	Anions by IC,
TMW47102022	J213-05	10-13-22	S3VM	Anions by IC,
TMW24102022	J213-06	10-13-22	S3VM	Anions by IC,
TMW49102022	J213-07	10-13-22	S3VM	Anions by IC,
MW24102022	J213-08	10-13-22	S3VM	Anions by IC,
BGMW11102022MS	J213-04M	10-13-22	S3VM	Anions by IC,
BGMW11102022MSD	J213-04S	10-13-22	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
		Analysis within 28 days for Bromide, Fluoride, Chloride and Sulfate	None. Holding times were met

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 2.1˚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:

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

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:

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

Completeness (EPA Method 9056A: Anions) =8/8X100=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 eight 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 eight (8) water samples were collected on 10-13-22. Samples were analyzed on 10-14-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Samples were analyzed and re-analyzed on 10-31-22, 11-01-22 and 11-03-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

4.12.2. 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 05-04-22, 08-09-22 and 10-18-22. 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 05-04-22, 08-09-22 and 10-18-22. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at

10-injections interval were analyzed on 10-14-22, 10-31-22, 11-01-22 and 11-03-22. A total of fourteen 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 the 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.

4.12.3. Quality Control Samples consisted of two method blanks, two sets of LCS/LCSD, MS/MSD and sample/sample duplicate. Sample BGMW11102022 was designated to be spiked as MS/MSD and sample/sample duplicate. Two more sets of MB and LCS/LCSD were analyzed with Chloride and Sulfate analysis on 10-31-22 and 11-03-22. 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.

Recoveries of MS/MSD were mostly within acceptable limits except for few anions as indicated in the table below:

Anion	BGMW11102022 MS%	BGMW11102022 MSD%	%Acceptance limits
Bromide	53*	51*	91-110
Nitrate	70*	69*	88-111
Nitrite	2*	2*	87-111
O-Phosphate	65*	69*	80-116
Sulfate	85*	92	87-112

*Outside Acceptance limits

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

4.12.5. Raw data was submitted for all requested field samples. Sample MW22D102022 (Lab ID#22J213-03) was designated 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 #22J213 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.

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J234 Analytical Data Package

Publication Date: 02-28-2023

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 2022 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 six (6) water samples were collected on 10-14-22. EMAX Laboratories received the samples on 10-15-22. 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 MW36D102022
(Lab ID#J234-03) was designated 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. Sample TMW39D102022 was selected to be spiked as MS/MSD. Recoveries and results of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision. Raw data for method blanks MS/MSD and LCS/LCSDs were also 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 (6 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 # 22J234) 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 six (6) water samples were collected on 10-14-22. EMAX Laboratories received the samples on 10-15-22.

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,
- 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 MW36D102022 was designated 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. Sample TMW39D102022 was selected by the lab to be spiked as MS/MSD. Raw data for MS/MSD with each set of 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#22J234 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
MW36S102022	J234-01	10-14-22	S3VM	Anions by IC,
TMW21102022	J234-02	10-14-22	S3VM	Anions by IC,
MW36D102022	J234-03	10-14-22	S3VM	Anions by IC,
TMW39D102022	J234-04	10-14-22	S3VM	Anions by IC,
TMW40D102022	J234-05	10-14-22	S3VM	Anions by IC,
TMW48102022	J234-06	10-14-22	S3VM	Anions by IC,
TMW39D102022MS	J234-04M	10-14-22	S3VM	Anions by IC,
TMW39D102022MSD	J234-04S	10-14-22	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 Analysis within 28 days for Bromide, Fluoride, Chloride and Sulfate	None. Holding times were met None. Holding times were met

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 5.6˚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) =6/6X100=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 six 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 six (6) water samples were collected on 10-14-22. Samples were analyzed on 10-15-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Samples were re-analyzed on 10-25-22, 10-26-22 and 10-27-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

4.12.2. 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 05-04-22 and 10-18-22. 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 05-04-22 and 10-18-22. Recoveries were all within

90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-15-22, 10-25-22, 10-26-22 and 10-27-22. A total of fourteen 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 the 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.

4.12.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD, MS/MSD and sample/sample duplicate. Sample TMW39D102022 was selected to be spiked as MS/MSD and sample/sample duplicate. Two more sets of MB and LCS/LCSD was analyzed with Chloride and Sulfate analysis on 10-25-22 and 10-26-22. 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.

Recoveries of MS/MSD were mostly within acceptable limits except for few anions as indicated in the table below:

Anion	TMW39D102022 MS%	TMW39D102022 MSD%	% Acceptance limits
Bromide	47*	48*	91-110
Nitrate	79*	77*	88-111
Nitrite	202*	202*	87-111
O-Phosphate	67*	69*	80-116

*Outside Acceptance limits

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

4.12.5. Raw data was submitted for all requested field samples. Sample MW36D102022 (Lab ID#22J234-03) was designated 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 #22J234 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.

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

Project No: Eco-18-1237

SDG #22J235 Analytical Data Package

Publication Date: 02-10-23

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 2022 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 seven (7) water samples were collected on 10-14-22. EMAX Laboratories received the samples on 10-15-22. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample MW36D102022(Lab ID# J235-04) was designated to be reviewed as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation review. No sample was designated to be spiked as MS/MSD on the chain of custody. Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision. Raw data for method blank and LCS/LCSD for each method 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, (both extraction and analysis), extraction logs, 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 methods of analysis:
EPA Method 3050B/8260C: Volatile Organics by GC-MS (7 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (5 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (6 samples)
EPA Method SW8332: Nitroglycerine and PETN (6 samples)
EPA Method 8081B: Organochlorine Pesticides (5 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (2 sample)

EPA Method 8151A: Chlorinated herbicides (2 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (4 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (3samples)
EPA Method 6850: Perchlorate (6 samples)

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 Methods (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 extraction and analytical holding times were met for each method and all the related samples. The deviations, if any, are discussed in Section 4.0 for each method.

The analytical data evaluated in this data validation report (SDG # 22J235) has met the data quality and usability requirement as defined in the data quality objectives. While very few analytical QC exceedances were observed, it was not significant for any data qualifiers. 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 DoD *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 seven (7) water samples were collected on 10-14-22. EMAX Laboratories received the samples on 10-15-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the 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 preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if submitted
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses 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. Next, selected analytical reports including QA/QC information was cross checked with raw data. The analysis and extraction sequence logs for each method were 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, surrogate recoveries, MS/MSD, and lab control samples (LCS/LCSD) 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 each analytical method requested on the chain of custody were submitted for all samples. One field sample MW36D102022 (Lab ID# J235-04) from this sample delivery group was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample was evaluated comprehensively. No sample was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank and LCS/LCSD was reviewed in detail. 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 regard to the requested method of analysis (collection to extraction and extraction 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
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated; if applicable
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards, and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

The following is a list of sample identifications and corresponding laboratory sample identification numbers:

Site Name: Fort Wingate, New Mexico

SDG#22J235 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW36S102022	22J235-01	10-14-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW39D102022	22J235-02	10-14-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
TMW21102022	22J235-03	10-14-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline; TPH as DRO Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
MW36D102022	22J235-04	10-14-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850 TPH Gasoline; TPH as DRO Polychlorinated Biphenyls Chlorinated Herbicides
TMW40D102022	22J235-05	10-14-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#22J235 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW48102022	22J235-06	10-14-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
QC14102022TB10	22J235-07	10-14-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 μg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20μg/L, (Benzidine=40μg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4μg/L
	Nitroglycerine & PETN	SW8330B	120μg/L
	Chlorinated Herbicides	SW8151A	1μg/L, (MCPA=40μg/L)
	Organochlorine Pesticides	SW8081B	0.1μg/L Methoxychlor =1.0μg/L Toxaphene =2.0μg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1μg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100μg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5μg/L,1μg/L,20μg/L,100μg/L,200μg/L
	Dissolved Mercury/Mercury	SW7470A	0.5μg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2μg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in nine ice preserved coolers.

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction, and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements 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"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	Not applicable
Anions by IC	Water	Analysis 48 hours from collection for Nitrate, Nitrite &Orthophosphate and 28-days for Bromide, Chloride, Fluoride and Sulfate	Not applicable
Mercury & Dissolved Mercury	Water	Collection to Analysis: 28 days	Not applicable

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 and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in nine ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 0.9˚C and as high as 5.6˚C upon arrival. All samples were received intact and 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 exactly the same way. 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:

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

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. The completeness objective is to obtain a sufficient amount 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:

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

Completeness (EPA Method 5030B/8260C: VOCs) =7/7X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =5/5X100=100%
Completeness (EPA Method 8330B: Explosives) =6/6X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =6/6X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =5/5X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =2/2X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =2/2X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 4/4X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =3/3X100=100%
Completeness (EPA Method 6850: Perchlorate) =6/6X100=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 seven water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS

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. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Seven water samples were collected on 10-14-22. Samples were analyzed on 10-26-22, 10-27-22and 10-28-22 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

Chain-of-custody was reviewed for documentation of sample information and method of analysis. Qualification notations, if any, will be summarized in result section; section 4.1.7.

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to generation of each initial calibration on

10-15-22, 10-17-22 and at the beginning of each analysis shift on 10-26-22 and 10-28-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to two sets of initial calibration using GC/MSD. Initial calibration curves were generated on 10-15-22 and 10-17-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table:

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 10-15-22	Average Response factor 10-17-22
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Bromoform	≥ 0.10	√	√
Chlorobenzene	≥ 0.50	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.045 & 0.036) and 2-Butanone (0.087 & 0.014).

However, recoveries were within the requirement of 70-130%. Calibration check compounds (CCCs) met the acceptance criteria for %RSD among the response factors calculated for each level. Table 4.1.3.2 lists the CCCs with method requirement limits for %RSD among response factors for initial calibration.

Table 4.1.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Response Factors %RSD 10-15-22	Response Factors %RSD 10-17-22
Vinyl chloride	-≤ 20%	√	√
1,1-Dichloroethene	-≤ 20%	√	√
Chloroform	-≤ 20%	√	√
1,2-Dichloropropane	-≤ 20%	√	√
Toluene	-≤ 20%	√	√
Ethyl benzene	-≤ 20%	√	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Each initial calibration curve was verified by a second source standard on10-15-22 and 10-17-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standard was analyzed at the beginning and end of each analysis shift on 10-26-22, and 10-28-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-26-22	Continuing cal. Response factors 10-28-22
		Opening & closing Daily St.	Opening &closing Daily St.
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. The calculated % difference between RFs from continuing calibration and average response factors from initial calibration is summarized in Table 4.1.4.2 for continuing calibration reports presented with the data package.

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-26-22)	%Deviation from Initial calibration (10-28-22)
		Opening & closing Daily St.	Opening & closing Daily St.
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list.

4.1.5. Quality Control samples reported consisted of two method blanks, and two sets of LCS/LCSD. No sample was designated on the chain of custody to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were all within the project acceptance limits for the entire compound list.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blanks presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method's acceptable limits.

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

4.1.7. Raw data was submitted for all samples. Sample MW36D102022 (Lab ID# 22J235-04) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

4.2. SVOC (EPA Method 3520C/8270D)

4.2.1. Technical Holding Times

Holding time requirement was met for all nine samples. Water samples were collected on 10-14-22, extracted on 10-21-22 and were analyzed on 11-02-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 11-02-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group.

Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table.

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-07-22
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Average response factors for the rest of target compounds were within method's recommended values.

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 20%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-07-22
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 03-07-22
Benzoic acid	0.9969
2,4-Dinitrophenol	0.9964
4-Nitrophenol	0.9971
Di-n-Octyl phthalate	0.9972

All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 11-02-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (11-02-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Percent difference between RFs from continuing calibration and average response factors from initial calibration passed the method's criteria as summarized in Table 4.2.4.2.

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (11-02-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for some compounds in closing daily standard, as listed in the table below:

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (11-02-22)	
		Opening Daily St.	Closing Daily St.
2,4-Dinitrophenol	≤ 20%	23.7*	√
Phenol	≤ 20%	√	20.7*
2-Methylphenol	≤ 20%	√	23.1*
Bis(2-Chloroisopropyl) ether	≤ 20%	√	27.7*
4-Methylphenol	≤ 20%	√	20.9*
4-Chloro-3-methylphenol	≤ 20%	√	24.8*

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (11-02-22)	
Fluoranthene	≤ 20%	√	25.6*
Bis(2-Ethylhexyl) phthalate	≤ 20%	√	21.2*

*Outside QC limit √ Denotes passing method acceptance limits

The compounds were not detected in the corresponding field samples. This should not affect the data quality.

4.2.5. Quality Control samples reported consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries for the LCS/LCSD reported were all within the project acceptance limits.

Recoveries of LCS/LCSD were used to evaluate both accuracy and precision. Four compounds failed maximum 20%RPD limits in LCS/LCSD; Hexachlorocyclopentadiene:22%, Caprolactam:22%, Benzaldehyde:26% and 1,2,4,5-Tetrachlorobenzene:32%. These compounds were not detected in any of the field samples.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.
Surrogate recoveries were all within the method's acceptable limits.

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

4.2.7. Raw data was submitted for all samples. Sample MW36D102022(Lab ID#J235-04) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.3. ORGANOCHLORINE PESTICIDES (EPA Method 3520C/ 8081B)

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for five water samples requested for this method. Water samples were collected on 10-14-22, extracted on 10-21-22 and analyzed on 10-24-22 within required holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 09-02-22, 09-03-22 and before sample analysis on 10-24-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 09-02-22 and 09-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 08-11-22 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene.

Retention time window width were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 08-11-22 and 09-12-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Performance of instrument was monitored by analysis of DDT and Endrin breakdown mixture. Before each continuing (daily) calibration standard, a mixture of DDT and Endrin was analyzed. Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of two continuing calibration standards were analyzed at 10-injections interval on 10-24-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. In the two continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) and %RPDs were within established QC limits.

Results and recoveries of QC samples were confirmed with the reported raw data.

Results for method blank were reviewed for each component and no organochlorine pesticide was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

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

4.3.6. Raw data was submitted for sample and all QC samples. Sample MW36D102022 (Lab ID#22J235-04) was designated as stage 3 data deliverable on the chain of custody. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. Polychlorinated Biphenyls (EPA Method 3520C/ 8082A)

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Two water samples were collected on 10-14-22, extracted on 10-21-22, and analyzed on 10-22-22, within holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 09-07-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

Retention time window width were established at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 09-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-22-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Results and recoveries of LCS/LCSD was used for both accuracy and precision. Percent recoveries (%R) were within the established QC limits.

Results for method blank was reviewed for each component and no target analyte was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

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

4.4.6. Raw data was submitted for all samples. Sample MW36D102022(Lab ID#J235-04) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Two water samples were collected on 10-14-22, extracted on 10-19-22 and analyzed on 10-21-22 and 10-22-22.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 10-03-22 and 10-04-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among

calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99819 was used for MCPP in column B.

Retention time windows were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 10-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-21-22 and 10-22-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were all less than 20%, except for the compounds summarized in the table below in the closing daily standard:

Target Compounds	**%Deviation From Initial calibration (Accept. Limit)**	**%Deviation from Initial calibration (10-21-22) Opening daily std.**		**%Deviation from Initial calibration (10-22-22) Closing daily std.**	
		Column A	Column B	Column A	Column B
MCPP	≤ 20%	√	25*	√	24*
Acifluorfen	≤ 20%	√	√	√	26*

*Outside QC limit

None of these compounds were detected in the field samples, and it should not affect the quality of data.

Results for surrogate recoveries and QC were all reported from channel A. Channel B was used for confirmation only.

4.5.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. All herbicides target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits

Results for method blank was reviewed for each component and no Herbicide was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

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

4.5.6. Raw data was submitted for all samples. Sample MW36D2022(Lab ID#J235-04) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Nitroaromatics by HPLC/UV (EPA Method 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six (6) water samples requested for this method. Water samples were collected on 10-14-22, extracted on 10-18-22 and analyzed on 10-19-22, within holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis. Positive results, if any, were confirmed with UHPLC equipped with different column (Kinetex- Biphenyl column).

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-25-22 and 08-26-22. Confirmation (Kinetex-Biphenyl column) was calibrated on 08-23-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

Retention time windows were established for each target analyte at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for primary column on 08-26-22 and confirmation column on 08-23-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of three continuing calibration standards were analyzed on 10-19-22 and 10-20-22, bracketing the analyses of samples and all the QC samples. Two more daily standards were also analyzed with confirmation column on 10-21-22. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

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

4.6.6. Raw data was submitted for all samples. Sample MW36D102022(Lab ID#J235-04) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation column (Biphenyl column).

4.7. Nitroglycerine and PETN by UHPLC/UV (EPA Method 8332)

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six (6) water samples requested for this method. Water samples were collected on 10-14-22, extracted on 10-18-22 and analyzed on 10-18-22 and 10-19-22 within the holding time requirements.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis.

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 08-24-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time windows were established for each target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 08-24-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-18-22 and 10-19-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

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

4.7.6. Raw data was submitted for all samples. Sample MW36D102022(Lab ID#J235-04) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.8. Total Petroleum hydrocarbons GRO (EPA Method 8015G)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for four water samples requested for this method. Water samples were collected on 10-14-22. Samples were analyzed on 10-21-22 and 10-22-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Sample was carried through the system by purge and trap.

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-06-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10). Retention times for further sample analyses was used for peak identification and integration range.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-06-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-21-22 and 10-22-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD. Raw data for QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

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

4.8.6. Raw data was submitted for all samples. Sample MW36D102022(Lab ID#J235-04) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for three water sample requested for this method. Water samples were collected on 10-14-22, extracted on 10-21-22 and analyzed on 10-25-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Heavier range of total petroleum hydrocarbons were extracted and introduced into system by direct injection.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-08-22. Calibration factor (area for each compound/concentration) was used to quantify diesel range hydrocarbons (TPH as DRO). A second set of initial calibration curve was generated for lighter TPHs (Jet Fuel) and heavier TPHs (motor oil). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time window width was established by analysis of a window defining hydrocarbon standard (C10-C40). Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-08-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

A total of two continuing calibration standards were analyzed on 10-25-22 at 10-injections interval, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) of LCS/LCSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

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

4.9.6. Raw data was submitted for all samples. Sample MW36D102022(Lab ID#J235-04) was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.10. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six water samples requested for this method. Water samples were collected on 10-14-22. Samples were analyzed on 10-18-22 within required holding time.

A High-Performance LC coupled with Mass Detector (HPLC/MS) was used for analysis.

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-21-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9960 (Perchlorate ion 83) and 0.9987 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

Retention time for each isotope (ion 83 and 85) at each calibration level was within 0.2 minutes required by the method.

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-21-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of seven daily standards were carried out on 10-18-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within
85-115% limit.

4.10.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established QC limits for both sets LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. No surrogate is used in this method.

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

4.10.6. Raw data was submitted for all samples. Sample MW36D102022(Lab ID#J235-04) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #22J235 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. 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).

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

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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22J236 Analytical Data Package

Publication Date: 02-13-23

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 2022 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 10-13-22. EMAX Laboratories received the samples on 10-15-22. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample MW22D102022(Lab ID# J236-03) was designated as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation review. Sample BGMW11102022 (Lab ID# J236-04) was designated to be spiked as MS/MSD on the chain of custody. Results and recoveries of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision. Raw data for method blank and LCS/LCSD and MS/MSD for each method 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, (both extraction and analysis), extraction logs, 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 methods of analysis:

EPA Method 3050B/8260C: Volatile Organics by GC-MS (9 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (7 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (8 samples)
EPA Method SW8332: Nitroglycerine and PETN (8 samples)
EPA Method 8081B: Organochlorine Pesticides (8 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (2 sample)

EPA Method 8151A: Chlorinated herbicides (2 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (4 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (3 samples)
EPA Method 6850: Perchlorate (8 samples)

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 Methods (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 extraction and analytical holding times were met for each method and all the related samples. The deviations, if any, are discussed in Section 4.0 for each method.

The analytical data evaluated in this data validation report (SDG # 22J236) has met the data quality and usability requirement as defined in the data quality objectives. While very few analytical QC exceedances were observed, it was not significant for any data qualifiers. 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 DoD *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 10-13-22. EMAX Laboratories received the samples on 10-15-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the 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 preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if submitted
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses 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. Next, selected analytical reports including QA/QC information was cross checked with raw data. The analysis and extraction sequence logs for each method were 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, surrogate recoveries, MS/MSD, and lab control samples (LCS/LCSD) 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 each analytical method requested on the chain of custody were submitted for all samples. One field sample MW22D102022 (Lab ID# J236-03) from this sample delivery group was designated as stage 3 data deliverable on the chain of custody. Raw data for this sample was evaluated comprehensively. Sample BGMW11102022 was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank and LCS/LCSD and MS/MSD was reviewed in detail. 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 regard to the requested method of analysis (collection to extraction and extraction 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
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated;
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards, and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

The following is a list of sample identifications and corresponding laboratory sample identification numbers:

Site Name: Fort Wingate, New Mexico

SDG#22J236 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW39102022	22J236-01	10-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850
TMW32102022	22J236-02	10-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
MW22D102022	22J236-03	10-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
BGMW11102022	22J236-04	10-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850
TMW47102022	22J236-05	10-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#22J36 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW24102022	22J236-06	10-13-22	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
TMW49102022	22J236-07	10-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
MW24102022	22J236-08	10-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Perchlorate by 6850
QC13102022 TB9	22J236-09	10-13-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
BGMW11102022MS	22J236-04M	10-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850
BGMW11102022MSD	22J236-04S	10-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine &PETN Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850

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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8330B	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in twelve ice preserved coolers.

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction, and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements 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"
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	NA
Anions by IC	Water	Analysis 48 hours from collection for Nitrate, Nitrite &Orthophosphate and 28-days for Bromide, Chloride, Fluoride and Sulfate	NA
Mercury & Dissolved Mercury	Water	Collection to Analysis: 28 days	NA

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 and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in twelve ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.3˚C and as high as 3.1˚C upon arrival. All samples were received intact and 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 exactly the same way. 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:

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

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. The completeness objective is to obtain a sufficient amount 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:

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

Completeness (EPA Method 5030B/8260C: VOCs) =9/9X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =7/7X100=100%
Completeness (EPA Method 8330B: Explosives) =8/8X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =8/8X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =8/8X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =2/2X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =2/2X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 4/4X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =3/3X100=100%
Completeness (EPA Method 6850: Perchlorate) =8/8X100=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 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS

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. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Nine water samples were collected on 10-13-22. Samples were analyzed on 10-26-22, 10-27-22 within method's requirement for holding time. (Water samples were preserved with hydrochloric acid).

Chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.1.7.

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 10-15-22 and at the beginning of analysis shift on 10-26-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 10-15-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table:

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 10-15-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.045) and 2-Butanone (0.087).

However, recoveries were within the requirement of 70-130%. Calibration check compounds (CCCs) met the acceptance criteria for %RSD among the response factors calculated for each level. Table 4.1.3.2 lists the CCCs with method requirement limits for %RSD among response factors for initial calibration.

Table 4.1.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Response Factors %RSD 10-15-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 10-15-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standard was analyzed at the beginning and end of analysis shift on 10-26-22 and 10-27-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-26-22 (Opening)	Continuing cal. Response factors 10-27-22 (Closing)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for

all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. The calculated % difference between RFs from continuing calibration and average response factors from initial calibration is summarized in Table 4.1.4.2 for continuing calibration reports presented with the data package.

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-26-22) Opening	%Deviation from Initial calibration (10-27-22) Closing
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list except for compounds in the table below for each daily standard:

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-26-22) %	%Deviation from Initial calibration (10-27-22) %
2-Chloroethylvinyl ether	≤ 20%	38.1*	√

*Outside control limits

This compound was not detected in the corresponding field samples. This should not affect the data quality.

4.1.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample BGMW11102022 (Lab ID# J236-04 and was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blanks presented with the data package, analyzed with samples did not show presence of any target compounds.
Surrogate recoveries were all within the method's acceptable limits.

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

4.1.7. Raw data was submitted for all samples. Sample MW22D102022 (Lab ID# 22J236-03) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

4.2. SVOC (EPA Method 3520C/8270D)

4.2.1. Technical Holding Times

Holding time requirement was met for all nine samples. Water samples were collected on 10-13-22, extracted on 10-20-22 and were analyzed on 11-10-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.
Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 11-10-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group.

Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table.

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-07-22
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Average response factors for the rest of target compounds were within method's recommended values.

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 20%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-07-22
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-07-22
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Least square linear regression was used for the following compounds where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 03-07-22
Benzoic acid	0.9969
2,4-Dinitrophenol	0.9964
4-Nitrophenol	0.9971
Di-n-Octyl phthalate	0.9972

All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 11-10-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (11-10-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Percent difference between RFs from continuing calibration and average response factors from initial calibration passed the method's criteria as summarized in Table 4.2.4.2.

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (11-10-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (11-10-22) I& II
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds from closing daily standard as listed in the table below:

Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (11-10-22) %	
		I	II
2,4-Dinitrophenol	≤ 20%	22.1*	√
Bis (2-Chloroisopropyl ether)	≤ 20%	√	21.0*

4.2.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample BGMW11102022 was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for the QC samples reported were mostly within the project acceptance limits, except for compounds listed in the table below:

Compound	BGMW11102022 (J236-04) MS%	BGMW11102022 (J236-04) MSD%	QC Limit%
2,4,5-Trichlorophenol	47*	√	53-123
2,4,6-Trichlorophenol	40*	√	50-125
2,4-Dichlorophenol	33*	√	47-121
2-Chlorophenol	22*	√	38-117
2-Nitrophenol	28*	√	47-123
Phenol	28*	√	50-130
2,6-Dichlorophenol	32*	√	50-118
Benzidine	23*	9*	30-160
2-Fluorophenol (surrogate)	17*	√	19-119
Phenol-d5 (surrogate)	31*	√	40-130

*Outside QC limit

Therefore, the recoveries of MSD and LCS/LCSD were used to evaluate the QC.
Quite a number of compounds failed maximum 20%RPD limits in MS/MSD. These compounds were not detected in any of the field samples.
Results and recoveries of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision for the rest of target list in this method.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.
Surrogate recoveries were all within the method's acceptable limits except for 2-Fluorophenol (17%) and Phenol-d5 (31%) as indicated in the above table.

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

4.2.7. Raw data was submitted for all samples. Sample MW22D102022(Lab ID#J236-03) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.3. ORGANOCHLORINE PESTICIDES (EPA Method 3520C/ 8081B)

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight water samples requested for this method. Water samples were collected on 10-13-22, extracted on 10-20-22 and analyzed on 10-22-22 within required holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 09-02-22, 09-03-22 and before sample analysis on 10-21-22 and 10-22-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 09-02-22 and 09-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 08-11-22 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene.

Retention time window width were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 08-11-22 and 09-12-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Performance of instrument was monitored by analysis of DDT and Endrin breakdown mixture. Before each continuing (daily) calibration a mixture of DDT and Endrin was analyzed. Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of four continuing calibration standards were analyzed at 10-injections interval on 10-21-22 and 10-22-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B.

In the four continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD and MS/MSD. Sample BGMW11102022 (Lab ID#J236-04) was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within established QC limits except for one compound as indicated in the table below:

Compound	BGMW11102022 (J236-04) MS%	BGMW11102022 (J236-04) MSD%	**QC Limit%**
Endrin	0*	12*	60-138

Therefore, result for this compound will be qualified as estimated value “UJ” in the parent sample.

Results and recoveries of QC samples were confirmed with the reported raw data.

Results for method blank were reviewed for each component and no organochlorine pesticide was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

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

4.3.6. Raw data was submitted for all samples. Sample MW22D102022(Lab ID#22J236-03) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. Polychlorinated Biphenyls (EPA Method 3520C/ 8082A)

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Two water samples were collected on 10-13-22, extracted on 10-20-22, and analyzed on 10-21-22, within holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 09-07-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

Retention time window width were established at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 09-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-21-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD and MS/MSD. Sample BGMW11102022 (Lab ID#J236-04) was designated to be spiked as MS/MSD. Results and recoveries of LCS/LCSD and MS/MSD was used for both accuracy and precision. Percent recoveries (%R) were within the established QC limits.

Results for method blank was reviewed for each component and no target analyte was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

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

4.4.6. Raw data was submitted for all samples. Sample MW39102022(Lab ID#J236-01) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Two water samples were collected on 10-13-22, extracted on 10-19-22 and analyzed on 10-21-22 and 10-22-22.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 10-03-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99819 was used for MCPP in column B.

Retention time windows were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 10-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-21-22 and 10-22-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels except for MCPP in column B (25% and 24% respectively).

Results for surrogate recoveries and QC were all reported from channel A. Channel B was used for confirmation only.

4.5.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample BGMW11102022 (Lab ID#J236-04) was designated to be spiked as MS/MSD. All herbicides target list was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within established QC limits

Results for method blank was reviewed for each component and no Herbicide was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

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

4.5.6. Raw data was submitted for all samples. Sample MW39102022(Lab ID#J236-01) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Nitroaromatics by HPLC/UV (EPA Method 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight (8) water samples requested for this method. Water samples were collected on 10-13-22, extracted on 10-18-22 and analyzed on 10-19-22 and 10-20022, within holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis. Positive results, if any, were confirmed with UHPLC equipped with different column (Kinetex- Biphenyl column).

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-25-22 and 08-26-22. Confirmation (Kinetex-Biphenyl column) was calibrated on 08-23-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

Retention time windows were established for each target analyte at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for both columns on 08-23-21 and 08-26-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of four continuing calibration standards were analyzed on 10-19-22 and 10-20-22, bracketing the analyses of samples and all the QC samples. Two daily standards were also analyzed with confirmation column on 10-21-22. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample BGMW11102021 (Lab ID#J236-04) was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

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

4.6.6. Raw data was submitted for all samples. Sample MW22D102022(Lab ID#J236-03) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation (Biphenyl column).

4.7. Nitroglycerine and PETN by UHPLC/UV (EPA Method 8332)

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight (8) water samples requested for this method. Water samples were collected on 10-13-22, extracted on 10-18-22 and analyzed on 10-18-22 and 10-19-22 within the holding time requirements.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis.

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 08-24-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time windows were established for each target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 08-24-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-18-22 and 10-19-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample BGMW11102021 (Lab ID#J236-04) was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

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

4.7.6. Raw data was submitted for all samples. Sample MW22D102022(Lab ID#J236-03) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.8. Total Petroleum hydrocarbons GRO (EPA Method 8015G)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for four water samples requested for this method. Water samples were collected on 10-13-22. Samples were analyzed on 10-21-22 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Sample was carried through the system by purge and trap.

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 08-04-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10). Retention times for further sample analyses was used for peak identification and integration range.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 08-04-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-21-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample BGMW11102022 (Lab ID#J236-04) was designated to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

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

4.8.6. Raw data was submitted for all samples. Sample MW22D102022(Lab ID#J236-03) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven water sample requested for this method. Water samples were collected on 10-13-22, extracted on 10-19-22 and analyzed on 10-25-22.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Heavier range of total petroleum hydrocarbons were extracted and introduced into system by direct injection.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-08-22. Calibration factor (area for each compound/concentration) was used to quantify diesel range hydrocarbons (TPH as DRO). A second set of initial calibration curve was generated for lighter TPHs (Jet Fuel) and heavier TPHs (motor oil). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

Retention time window width was established by analysis of a window defining hydrocarbon standard (C10-C40). Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-08-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

A total of three continuing calibration standards were analyzed on 10-25-22 at 10-injections interval, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample BGMW11102022 (Lab ID#J236-04) was designated for MS/MSD. Percent recoveries (%R) of LCS/LCSD and MS/MSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

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

4.9.6. Raw data was submitted for all samples. Sample MW22D102022(Lab ID#J236-03) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.10. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight water samples requested for this method. Water samples were collected on 10-13-22. Samples were analyzed on 10-18-22 within required holding time.

A High-Performance LC coupled with Mass Detector (HPLC/MS) was used for analysis.

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-21-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9960 (Perchlorate ion 83) and 0.9987 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

Retention time for each isotope (ion 83 and 85) at each calibration level was within 0.2 minutes required by the method.

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-21-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of six daily standards were carried out on 10-18-22 and 10-19-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and one set of MS/MSD. Sample BGMW11102022 (Lab ID#J236-04) was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for MS/MSD and LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. No surrogate is used in this method.

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

4.10.6. Raw data was submitted for all samples. Sample MW22D102022(Lab ID#J236-03) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #22J236 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

6.0 REFERENCES

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