Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #21J023 Analytical Data Package

Publication Date: 01-27-2022

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 10-04-21. EMAX Laboratories received the samples on 10-05-21. 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 MW32102021 (Lab ID#J023-01) was selected to be reviewed as stage 3 deliverable. Raw data for this sample was compared to the reported summary table and went through comprehensive data validation. Sample TMW26102021 was designated to be spiked as MS/MSD. Raw data for this sample together with method blank and LCS/LCSD 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 initial sample analysis. However, two samples (TMW26102021 and TMW51102021) were analyzed for Fluoride, Bromide, and sulfate one day past 28-day required holding time. The deviations, are discussed in Section 4.0 for this method.

The SDG # 21J023 analytical data evaluated in this data validation report 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 10-04-21. EMAX Laboratories received the samples on 10-05-21.

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 MW32102021 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. Sample TMW26102021 was designated to be spiked as MS/MSD on the chain of custody. Raw data for this sample together with other QC samples in was also 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 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;
- LCSs 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#21J023 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
MW32102021	J023-01	10-04-21	S3VM	Anions by IC,
BGMW07102021	J023-02	10-04-21	S3VM	Anions by IC,
BGMW03102021	J023-03	10-04-21	S3VM	Anions by IC,
BGMW08102021	J023-04	10-04-21	S3VM	Anions by IC,
TMW26102021	J023-05	10-04-21	S3VM	Anions by IC,
TMW51102021	J023-06	10-04-21	S3VM	Anions by IC,
TMW26102021MS	J023-05M	10-04-21	S3VM	Anions by IC,
TMW26102021MSD	J023-05S	10-04-21	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 on 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 Except for two samples*

*Samples TMW26102021 and TMW51102021 were analyzed for Bromide, Fluoride and Sulfate one day past 28-day 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 3.5˚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 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 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, 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 six water samples requested for this method. Water samples were collected on 10-04-21. Samples were analyzed on 10-05-21 and 10-06-21 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Samples for Bromide, fluoride, Chloride, and Sulfate were analyzed and/or re-analyzed with dilution on 11-01-21 and 11-02-21within 28-day holding time. All initial analysis for Bromide, Chloride, Sulfate and fluoride were within 28-day holding time. However, samples TMW26102021 and TMW51102021 were re-analyzed one day past required 28-day holding time for Bromide, fluoride and Sulfate. Since exceedance for holding time is less than 2×HT requirement, the positive results of these anions would be qualified as estimated value ("J") and non-detect results are qualified as "UJ" for these two samples.

4.1.2. Initial and continuing calibration: Anions such as Bromide, Chloride, Fluoride, 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 on the basis of retention time as compared to reference standards.

Two instruments were initially calibrated with nine calibration levels (0.05-20mg/L) on 08-12-21 and 10-21-21 and 11-15-21. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout the analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. 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 08-12-21 and 10-21-21 and 11-15-21. Recoveries were all within 90-110% of expected true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-05-21, 10-06-21, 11-01-21, 11-02-21, 11-17-21 and 11-18-21. A total of seven continuing calibration standards were initially analyzed on 10-05-21 and 10-06-21. Later, twelve more continuing calibration standards were analyzed with reference to separate initial calibrations on 11-01-21, 11-02-21, 11-17-21 and 11-18-21. 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.

4.1.3. Quality Control Samples consisted of one method blank and one set of LCS/LCSD, MS/MSD and sample/sample duplicate for initial analysis of all anions. Sample TMW26102021was designated to be spiked as MS/MSD and sample/sample duplicate analysis. A second and third set of quality control samples (MB and LCS/LCD) was analyzed with re-analysis/dilutions of samples for Bromide, Chloride, Fluoride and Sulfate. Recoveries of LCS/LCSDs were all within 90-110 % of spiked values for each anion. Recoveries of MS/MSD were mostly within acceptable limits except for some anions as indicated in the table below:

Anion	TMW26102021 MS%	TMW26102021 MS%	% Acceptance limits
Nitrite	85*	85*	88-111
Orthophosphate	65*	66*	80-116
Bromide	71*	72*	91-110
Chloride	72*	96	87-111

Therefore, the positive results will be qualified as estimated value (“J”) and as (“UJ”) for non-detect results in the parent sample for these anions. Percent RPDs were less than 20% for LCS/LCSD and MS/MSD. Sample TMW26102021 was also used for sample/sample duplicate analysis. All the recoveries and % differences were all within acceptance limits.

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

4.1.5. Raw data was submitted for all requested field samples. Sample MW32102021 was selected to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met. Due to high concentrations of Sulfate and Chloride, samples were analyzed with high dilution factors on 11-01-21 and 11-02-21 within 28-day holding time. However, as mentioned in section 4.1.1, samples TMW26102021 and TMW51102021 were re-analyzed one day past required 28-day holding time for Sulfate, fluoride and Bromide. Sample TMW26102021 was run with more dilution on 11-17-21. Since exceedance for holding time is less than 2 times required 28- day hooding time (<½HT), the positive results for these anions would be qualified as estimated value (“J”) and non-detect results are qualified as “UJ” for these two samples.

5.0 CONCLUSION

SDG #21J023 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 #21J037 Analytical Data Package

Publication Date: 01-24-22

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 ten (10) water samples were collected on 10-04-21. EMAX Laboratories received the samples on 10-06-21. 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 TMW19102021(Lab ID# J037-08) 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. Sample TMW26102021(Lab ID# J037-09) was designated to be spiked as MS/MSD on the chain of custody. Raw data for this sample together with method blank and LCS/LCSD for each method 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, (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 (10 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (8 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (9 samples)
EPA Method SW8332: Nitroglycerine and PETN (9 samples)
EPA Method 8081B: Organochlorine Pesticides (5 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 (9 samples)
EPA Method SW6020A: Dissolved and total Metals by ICP-MS (9 samples)
EPA Method 7470A: Mercury & Dissolved Mercury (9 samples)

The analytical results, QC results, initial calibration summary tables and initial calibration verification standards (ICV) 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 # 21J037 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 ten (10) water samples were collected on 10-04-21. EMAX Laboratories received the samples on 10-06-21.

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 TMW19102021 (EMAX ID #J037-08) from this sample delivery group was designated for stage 3 data review. Raw data for this sample was evaluated comprehensively. Sample TMW26102021 (EMAX ID #J037-09) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with other QC samples in this report, including LCS/LCSD, was also 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#21J037 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW32102021	21J037-01	10-04-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850
QC04102021TB1	21J037-02	10-04-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
BGMW07102021	21J037-03	10-04-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850
TMW16102021	21J037-04	10-04-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
TMW16102021D	21J037-05	10-04-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#21J037 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
BGMW03102021	J037-06	10-04-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
BGMW08102021	J037-07	10-04-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850
TMW19102021	J037-08	10-04-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850
TMW26102021	J037-09	10-04-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
TMW51102021	J037-10	10-04-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls

Site Name: Fort Wingate, New Mexico

SDG#21J037 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW26102021MS	J037-09	10-04-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
TMW26102021MSD	J037-09	10-04-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS 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	SW8332	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
	Perchlorate	SW6850	0.5µg/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	None. Holding times were met
Mercury & Dissolved Mercury	Water	Collection to Analysis: 28 days	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 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 1.7˚C and as high as 5.5˚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) =10/10X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =8/8X100=100%

Completeness (EPA Method 8330B: Explosives) =9/9X100=100%

Completeness (EPA Method 8332: Nitroglycerine & PETN) =9/9X100=100%

Completeness (EPA Method 8081B: Organochlorine pesticides) =5/5X100=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) =9/9X100=100%

Completeness (EPA Method 7470A: Mercury & Dissolved Mercury) =9/9X100=100%

Completeness (EPA Method 6020A: Dissolved and Total Metals) =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 ten 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 **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury and Dissolved Mercury by Cold Vapor

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. Ten water samples were collected on 10-04-21. Samples were analyzed on 10-08-21, 10-11-21 and 10-12-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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 4.1.7.

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 07-23-21 and at the beginning of each analysis shift on 10-08-21, 10-11-21 and 10-12-21. 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 07-23-21. 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 07-23-21
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.

Average response factors for all target compounds were within method's recommended values, except for Acetone (0.036) and 2-Butanone (0.065). However, recoveries were all within 70-130% requirement.

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 07-23-21
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 07-24-21. 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 end of each analysis shift on 10-08-21, 10-11-21 and 10-12-21. Prior to each continuing calibration, **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 requirements 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 10-08-21 (I& II)	Continuing cal. Response factors 10-11-21 (I& II)	Continuing cal. Response factors 10-12-21 (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 (10-08-21) I&II	%Deviation from Initial calibration (10-11-21) I&II	%Deviation from Initial calibration (10-12-21) 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 the initial calibration was less than 20 percent for the rest of target list, except for Trichloroethene (43.6%) in the closing daily standard run on 10-11-21 and Chloromethane (21.0) in the closing daily standard run on 10-12-21. This should not affect data quality.

4.1.5. Quality Control samples reported consisted of three method blanks, three sets of LCS/LCSD and MS/MSD. Sample TMW26102021 (lab ID #J037-09) was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for MS/MSD and 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: Sample TMW16102021 was identified as field duplicate of TMW16102021D. Both sample and corresponding field duplicate sample were reported as non-detected for all Volatile Organic Compounds list.

4.1.7. Raw data was submitted for all samples. Sample TMW19102021 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 samples. Eight water samples were collected on 10-04-21, extracted on 10-11-21 and were analyzed on 10-13-21 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 calibrations on 08-12-21, 08-13-21 and at the beginning of analysis shift on 10-13-21. 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 08-12-21 and 08-13-21. 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 08-12-21
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 08-12-21
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)	Response Factors %RSD (Method limit)	Accepted Response Factors 08-12-21
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) 08-12-21
Benzoic acid	0.9991
2,4-Dinitrophenol	0.9993
4-Nitrophenol	1.00
4,6-Dinitro-2-Methylphenol	0.9983
Benzidine	0.9994
3,3-dimethylbenzidine	0.9999

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 08-12-21 and 08-13-21. 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 except 4,6-Dinitro-2-methylphenol (25.0%). Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-13-21. 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 (10-13-21) 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 nearly 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 (10-13-21) 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 listed below in the closing continuing calibration standard:

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (10-13-21) II
2,3,5,6-Tetrachlorophenol	≤ 20	20.8*

*Exceeded 20% maximum allowed difference in the closing daily standard

This deviation should not affect quality of the presented data.

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 for the entire compound list in LCS/LCSD. Recoveries of LCS/LCSD was used to evaluate accuracy and precision for 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 the surrogates listed in the samples below:

Surrogate	BGMW08102021 (21J037-07)	TMW19102021 (21J037-08)	**QC Limit%**
2-Fluorophenol	11%*	10%*	19-119
Phenol-D5	31%*	25%*	40-130
2,4,6-Tribromophenol	√	28%*	43-140

*Outside control limits

This was attributed to matrix interference in the case narrative.

4.2.6. **Field duplicate sample** and its associated sample: Sample TMW16102021 was identified as field duplicate of TMW16102021D. Both sample and corresponding field duplicate sample were reported as non-detected for the entire Semi-Volatile Compound list.

4.2.7. Raw data was submitted for all samples. Sample TMW19102021 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 water samples requested for this method. Five (5) water samples were collected on 10-04-21, extracted on 10-08-21 and analyzed on 10-27-21 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 08-23-21 and before sample analysis on 10-27-21. 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 08-23-21. 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 08-14-21 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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 08-14-21 and 08-24-21. 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 10-27-21, 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, %D for Endosulfan sulfate failed the maximum 20% difference in two of the last daily standards analyzed (42% and 31% respectively) in channel B. Few compounds failed maximum 20% difference in channel A. Since exceedances were all high biased and samples were all reported as non-detect, this does not affect the data quality. 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. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits except for one compound shown in the table below:

Surrogate	LCS	LCSD	**QC Limit%**
Endosulfan sulfate	150%*	154%*	62-133

*Outside control limits

This should not affect the quality of data; LCS/LCSD recoveries were biased high and this compound was not detected in any of the field samples in the batch.

Raw data for the QC samples were matching the reported values.

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 for this method.

4.3.6. Raw data was submitted for all samples. Sample TMW51102021 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 10-04-21, extracted on 10-08-21, and analyzed on 10-15-21 and 10-16-21.

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 10-14-21. 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 10-14-21. 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 were injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-15-21 and 10-16-21, 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 method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD, therefore, the 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 collected for this method.

4.4.6. Raw data was submitted for all samples. Sample TMW51102021 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 10-04-21, extracted on 10-11-21 and analyzed on 10-12-21.

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 09-17-21. 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.

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 09-17-21. 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 10-12-21, 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 compounds from channels B. At least six compounds exceeded 20% limit in column A.

Results for surrogate recoveries and QC were all reported from channel B. Channel A 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 for this method.

4.5.6. Raw data was submitted for all samples. Sample TMW51102021 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 nine (9) water samples requested for this method. Water samples were collected on 10-04-21, extracted on 10-08-21 and analyzed on 10-12-21 and 10-13-21, 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 08-24-21. 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 08-24-21. 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 10-12-21 and 10-13-21, 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 and MS/MSD. Sample TMW26102021 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 TMW16102021 was identified as field duplicate of sample TMW16102021D. No explosive target compound was detected in each sample and field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample TMW19102021 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 nine (9) water samples requested for this method. Water samples were collected on 10-04-21, extracted on 10-07-21 and analyzed on 10-08-21.

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. Confirmation column was calibrated on 10-23-21. 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 10-08-21, bracketing the analyses of samples and all the QC samples. Two daily standards were also analyzed with confirmation column on 10-19-21 and 10-20-21. 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 TMW26102021 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 TMW16102021 was identified as field duplicate of sample TMW16102021D. No explosive target compound was detected in sample and field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample TMW19102021 was designated to be reviewed as stage 3 data deliverable. Raw data of 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. 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 water samples requested for this method. Three (3) water samples were collected on 10-04-21. Samples were analyzed on 10-07-21 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 09-23-21. 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 09-23-21. 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 10-07-21, 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 collected for this method.

4.8.6. Raw data was submitted for all samples. Sample TMW51102021 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 water sample requested for this method. Two (2) water samples were collected on 10-04-21, extracted on 10-11-21 and analyzed on 10-12-21.

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 08-12-21. 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 08-12-21. 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 10-12-21 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 collected for this method.

4.9.6. Raw data was submitted for all samples. Sample TMW51102021 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 all sample requested for this method. Nine (9) water samples were collected on 10-04-21. Samples were analyzed on 10-12-21 and 10-13-21.

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 09-21-21. 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 0.999 (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 09-21-21. 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 10-12-21 and 10-13-21, 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 TMW26102021 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 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 TMW16102021 was identified as field duplicate of TMW16102021D. No perchlorate was detected in each sample and associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample TMW19102021 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. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.11.1. 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 10-04-21. Samples were prepared (digested) for both total and dissolved metals on 10-08-21. Samples and QC samples were analyzed on 11-01-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 11-01-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method's acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. The correlation coefficient of at least 0.999 was calculated to show the linearity of calibration curve for each metal. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

Metals	Concentration µg/L
Se, Pb, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Mn, Ni, Ag, Tl, V, and Zn	50,100,500,1000
Al, Fe, Ca, Mg, Na, and K	50,000

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of ±20% of the true value for the analytes incorporated in each solution.

4.11.3. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW26102021 was spiked as MS/MSD. Recoveries of LCS/LCSD were all within the acceptance limit of 80-120% for both total and dissolved metals. Recoveries of MS/MSD were mostly acceptable except the few metals as listed in the table below.

ANALYTE	Total Metals			Dissolved Metals		
	TMW26102021 MS%	TMW26102021 MSD%	QC Limit%	TMW26102021 MS%	TMW26102021 MSD%	QC Limit%
Aluminum	√	√	84-117	√	√	84-117
Antimony	√	√	85-117	√	√	85-117
Arsenic	√	√	84-116	√	√	84-116
Barium	√	√	86-114	√	√	86-114
Beryllium	√	√	83-121	√	√	83-121

ANALYTE	Total Metals			Dissolved Metals		
	TMW26102021 MS%	TMW26102021 MSD%	**QC Limit%**	TMW26102021 MS%	TMW26102021 MSD%	**QC Limit%**
Cadmium	√	√	87-115	√	√	87-115
Calcium	√	√	87-118	100	120*	87-118
Chromium	√	√	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	√	√	85-118
Iron	√	√	87-118	√	√	87-118
Lead	89	87*	88-115	88	87*	88-115
Magnesium	√	√	83-118	√	√	83-118
Manganese	√	√	87-115	√	√	87-115
Nickel	√	√	85-117	√	√	85-117
Potassium	√	√	85-115	√	√	85-115
Selenium	√	√	80-120	√	√	80-120
Silver	√	√	85-116	√	√	85-116
Sodium	867*	733*	85-117	-100*	-633*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

As a result, all the positive results for parent sample will be qualified as estimated value “J”. This was attributed to high dilution factor and matrix interference in the case narrative.

Method blank results for metals were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard. Sample TMW26102021 was used for serial dilution for both total and dissolved metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis. The same sample was used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total and dissolved metals.

4.11.4. Field duplicate sample and its associated sample: Sample TMW16102021 was identified as field duplicate of TMW16102021D. Results for sample/sample duplicate is summarized in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW16102021 µg/L	TMW16102021D µg/L	% RPD	TMW16102021 µg/L	TMW16102021D µg/L	% RPD
Aluminum	530	530	<1	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	24	22	8.69	11	10	9.52
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	6100	5900	3.33	4000	3900	2.53
Chromium	8.0	6.6	19.2	U	U	--
Cobalt	1.2J	1.2J	<1	U	U	--
Copper	21	19	10	U	3.9J	200
Iron	380J	370J	2.66	U	U	--
Lead	1.4J	1.2J	15.4	U	U	--
Magnesium	680	670	1.48	430J	440J	2.30
Manganese	30	29	3.39	4.3J	3.7J	15.0
Nickel	78	76	2.60	18	16	11.8
Potassium	690J	690J	<1	610J	620J	1.63
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	500000	500000	<1	450000	440000	2.25
Thallium	U	U	--	U	U	--
Vanadium	5.7	5.5	3.57	4.1J	4.0J	2.47
Zinc	36J	36J	<1	U	U	--

4.11.5. Raw data was submitted for all samples. Sample TMW19102021 was designated to be reviewed as stage 3 data deliverable. Raw data of 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.

4.12. MERCURY by COLD VAPOR (Mercury &Dissolved Mercury); EPA Method 7470A

4.12.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for nine (9) water samples requested for this method. Water samples were collected on 10-04-21. Samples were prepared (digested) and analyzed for Mercury and dissolved Mercury on 10-14-21. All samples were preserved and filtered for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of each analysis day on 10-14-21. The correlation coefficient of at least 0.999860 was calculated to show the linearity of calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

Initial calibration verification and Continuing calibration verification standards for mercury was within the acceptable range (90-110% of the spiked value).

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of method blank, LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Sample TMW26102021 was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW26102021 was also used for serial dilution. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample TMW16102021 was identified as field duplicate of TMW16102021D. No Mercury was detected in sample and corresponding field duplicate sample for Mercury and dissolved Mercury.

4.12.5. Raw data was submitted for all samples. Sample TMW19102021 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 raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #21J037 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 #21J038 Analytical Data Package

Publication Date: 01-24-22

By
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TABLE OF CONTENTS

EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 10-05-21. EMAX Laboratories received the samples on 10-06-21. 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 MW01102021(Lab ID# J038-01) 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. 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 (9 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (11 samples)
EPA Method SW8332: Nitroglycerine and PETN (11 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 (11 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (11 samples)

EPA Method 7470A: Mercury & Dissolved Mercury (11 samples)
EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate (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 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 # 21J038 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 twelve (12) water samples were collected on 10-05-21. EMAX Laboratories received the samples on 10-06-21.

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 MW01102021 (EMAX ID #J038-01) from this sample delivery group was designated for stage 3 data review. 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; only for total metals
- 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#21J038 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW01102021	21J038-01	10-05-21	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC; 9056A
TMW44102021	21J038-02	10-05-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; 9056A
MW02102021	21J038-03	10-05-21	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Perchlorate by 6850 Anions by IC; 9056A
BGMW01102021	21J038-04	10-05-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; 9056A

Site Name: Fort Wingate, New Mexico

SDG#21J038 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW46102021	J038-05	10-05-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Anions by IC; 9056A
QC05102021TB2	J038-06	10-05-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
MW28102021	J038-07	10-05-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC; 9056A
TMW58102021	J038-08	10-05-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC; 9056A

Site Name: Fort Wingate, New Mexico

SDG#21J038 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW29102021	J038-09	10-05-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC; 9056A
BGMW02102021	J038-10	10-05-21		VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; 9056A
TMW18102021	J038-11	10-05-21		VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; 9056A
QC05102021EB1	J038-12	10-05-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC; 9056A

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	SW8332	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	0.5μg/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	None. Holding times were met
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	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 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 1.0˚C and as high as 5.8˚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:

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

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) =10/10X100=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) =11/11X100=100%
Completeness (EPA Method 7470A: Mercury & Dissolved Mercury) =11/11X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =11/11X100=100%
Completeness (EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate) =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
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury & Dissolved Mercury by Cold Vapor
EPA **Method 9056A** for Anions 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 twelve samples. Water samples were collected on 10-05-21. Samples were analyzed on 10-13-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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 4.1.7.

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 08-18-21 and at the beginning of analysis shift on 10-13-21. 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 08-18-21. 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 08-18-21
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.

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

Target Analytes	Least Square Linear Regression (CCF) 08-18-21
Acetone	0.9989

Average response factors for all target compounds were within method's recommended values, except for: Acetone (0.031) and 2-Butanone (0.012). 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 08-18-21
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 08-19-21. 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 10-13-21. 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 10-13-21 (I)	Continuing cal. Response factors 10-13-21 (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 (10-13-21) I	%Deviation from Initial calibration (10-13-21) 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 the initial calibration was less than 20 percent for the rest of target list, except for Methyl acetate (28.6%) in the closing daily standard run on 10-13-21. This should not affect the data quality.

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: No field duplicate sample was assigned to this sample delivery group.

4.1.7. Raw data was submitted for all samples. Sample MW01102021 (Lab ID# 21J038-01) 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. Traces of Bromodichloromethane, Chloroform and Dibromochloromethane was detected in the equipment blank. However, these compounds were not detected in any of the field samples.

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 10-05-21, extracted on 10-11-21 and were analyzed on 10-13-21 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 08-12-21, 08-13-21 and at the beginning of analysis shift on 10-13-21. 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 08-12-21 and 08-13-21. 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 08-12-21
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 08-12-21
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) 08-12-21
Benzoic acid	0.9991
2,4-Dinitrophenol	0.9993
4-Nitrophenol	1.00
4,6-Dinitro-2-Methylphenol	0.9983
Benzidine	0.9994
3,3-dimethylbenzidine	0.9999

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 08-12-21 and 08-13-21. 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 except for 4,6-Dinitro-2-methylphenol (25.0%). Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on10-13-21. 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 (10-13-21) 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 nearly 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 (10-13-21) 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 (10-13-21) 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 the compounds listed below in the closing continuing standard:

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (10-13-21) II
2,3,5,6-Tetrachlorophenol	≤ 20	20.8*

*Exceeded 20% maximum allowed difference in the closing daily standard

This deviation should not affect quality of the presented data.

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 for the entire compound list in LCS/LCSD. Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision for 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 sample was collected with this sample delivery group.

4.2.7. Raw data was submitted for all samples. Sample MW28102021(Lab ID#21J038-07) 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 all ten water samples requested for this method. Water samples were collected on 10-05-21, extracted on 10-12-21 and analyzed on 10-30-21 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 08-23-21 and before sample analysis on 10-30-21. 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 08-23-21. 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 08-14-21 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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 08-14-21 and 08-24-21. 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 10-30-21, 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, %D for Delta BHC failed the maximum 20% difference in the closing daily standard (24% in channel 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. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits except for one compound shown in the table below:

Surrogate	LCS	LCSD	**QC Limit%**
Endosulfan sulfate	149*%	143*%	62-133

* Exceeded acceptable QC limits

Recoveries of LCS/LCSD were biased high and this compound was not detected in any of the field samples, therefore, this should not affect the quality of data.

Raw data for the QC samples were matching the reported values.

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 assigned to this sample delivery group.

4.3.6. Raw data was submitted for all samples. Sample MW01102021(Lab ID#21J038-01) 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.

4.4. 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 10-05-21, extracted on 10-12-21, and analyzed on 10-16-21.

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 10-14-21. 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 10-14-21. 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 were injected at single point for identification only.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-15-21 and 10-16-21, 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 sample delivery group.

4.4.6. Raw data was submitted for all samples. Sample MW28102021(Lab ID#21J038-07) 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 10-05-21, extracted on 10-11-21 and analyzed on 10-12-21.

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 09-17-21. 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.

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 09-17-21. 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 10-12-21, 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 compounds from channels B. At least six compounds exceeded 20% limit in column A.

Results for surrogate recoveries and QC were all reported from channel B. Channel A 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 sample delivery group.

4.5.6. Raw data was submitted for all samples. Sample MW28102021 (Lab ID#21J038-07) 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 10-05-21, extracted on 10-07-21 and analyzed on 10-07-21 and 10-08-21, 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 08-23-21 and 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 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 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% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of three continuing calibration standards were analyzed on 10-07-21 and 10-08-21, 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 only. No sample was designated to be spiked as MS/MSD. The entire 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 assigned to this sample delivery group.

4.6.6. Raw data was submitted for all samples. Sample MW01102021(Lab ID#21J038-01) was designated to be reviewed as stage 3 data deliverable. Raw data of 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. All positive results, if any were confirmed with confirmation 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 10-05-21, extracted on 10-07-21 and analyzed on 10-08-21.

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 10-08-21, 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 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 within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** No field duplicate sample was assigned to this sample delivery group.

4.7.6. Raw data was submitted for all samples. Sample MW01102021(Lab ID#21J038-01) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with all 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 10-05-21. Samples were analyzed on 10-07-21 and 10-08-21 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 09-23-21. 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 09-23-21. 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 10-07-21 and 10-08-21, 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 to this sample delivery group.

4.8.6. Raw data was submitted for all samples. Sample MW01102021(Lab ID#21J038-01) 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 10-05-21, extracted on 10-11-21 and analyzed on 10-12-21.

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 08-12-21. 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 08-12-21. 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 10-12-21 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 sample delivery group.

4.9.6. Raw data was submitted for all samples. Sample MW01102021(Lab ID#21J038-01) 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 eleven samples requested for this method. Water samples were collected on 10-05-21. Samples were analyzed on 10-12-21 and 10-13-21.

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 09-21-21. 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 0.999 (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 09-21-21. 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 10-12-21 and 10-13-21, 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 only. No sample was designated to be spiked as MS/MSD. Perchlorate was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established QC limits 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 assigned to this sample delivery group.

4.10.6. Raw data was submitted for all samples. Sample MW01102021 (Lab ID#21J038-01) 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. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.11.1. 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 10-05-21. Samples were prepared (digested) for both total and dissolved metals on 10-08-21. Samples and QC samples were analyzed on 11-01-21 and 11-02-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of each analysis day on 11-01-21 and 11-02-21. Tune performance report was generated at the start of each analysis day, before initial calibration. It was within method’s acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard was used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

Metals	Concentration μg/L
Se, Pb, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Mn, Ni, Ag, Tl, V, and Zn	50,100,500,1000
Al, Fe, Ca, Mg, Na, and K	50,000

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of ±20% of the true value for the analytes incorporated in each solution.

4.11.3. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD for total metals. Sample MW01102021 was spiked as MS/MSD for total metals only. Recoveries of LCS/LCSD were all within the acceptance limit of 80-120% for both total and dissolved metals. Recoveries of MS/MSD were mostly acceptable except the few metals as listed in the table below. As a result, all the positive results will be qualified as estimated value “J” for these metals in parent sample . This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	MW01102021 MS%	MW01102021 MSD%	QC Limit%	NA	NA	QC Limit%
Aluminum	104	118*	84-117	NA	NA	84-117
Antimony	√	√	85-117	NA	NA	85-117
Arsenic	√	√	84-116	NA	NA	84-116
Barium	√	√	86-114	NA	NA	86-114
Beryllium	√	√	83-121	NA	NA	83-121
Cadmium	√	√	87-115	NA	NA	87-115
Calcium	√	√	87-118	NA	NA	87-118
Chromium	√	√	85-116	NA	NA	85-116
Cobalt	√	√	86-115	NA	NA	86-115
Copper	√	√	85-118	NA	NA	85-118
Iron	101	124*	87-118	NA	NA	87-118
Lead	√	√	88-115	NA	NA	88-115
Magnesium	104	121*	83-118	NA	NA	83-118
Manganese	105	133*	87-115	NA	NA	87-115

ANALYTE	Total Metals			Dissolved Metals		
	MW01102021 MS%	MW01102021 MSD%	**QC Limit%**	NA	NA	**QC Limit%**
Nickel	√	√	85-117	NA	NA	85-117
Potassium	√	√	85-115	NA	NA	85-115
Selenium	√	√	80-120	NA	NA	80-120
Silver	√	√	85-116	NA	NA	85-116
Sodium	467*	-33*	85-117	NA	NA	85-117
Thallium	√	√	82-116	NA	NA	82-116
Vanadium	√	√	86-115	NA	NA	86-115
Zinc	√	√	83-119	NA	NA	83-119

*Outside control limits

Method blank results for metals were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard. Sample MW01102021 was used for serial dilution for total metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis. The same sample was used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total metals.

4.11.4. Field duplicate sample and its associated sample: No field duplicate was assigned to this sample delivery group.

4.11.5. Raw data was submitted for all samples. Sample MW01102021 (Lab ID#21J038-01) was designated to be reviewed as stage 3 data deliverable. Raw data of 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.

4.12. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.12.1. 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 10-05-21. Samples were prepared (digested) and analyzed for Mercury and dissolved Mercury on 10-14-21. All samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 10-14-21. The correlation coefficient of at least 0.999860 was calculated to show the linearity of calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

Initial calibration verification and Continuing calibration verification standards for mercury was within the acceptable range (90-110% of the spiked value).

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of on method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD for Mercury and dissolved Mercury. Recoveries of LCS/LCSD and were within acceptable range of 82-119%. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this sample delivery group.

4.12.5. Raw data was submitted for all samples. Samples MW01102021 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 raw data, agreed with all the results reported in data summary reports.

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

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of eleven water samples requested for this method. Water samples were collected on 10-05-21. Samples were analyzed on 10-06-21 and 10-07-21 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Some samples were re-analyzed on 11-01-21 for Chloride and Sulfate at higher dilutions within 28-day holding times.

4.13.2. 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 on the basis of retention time as compared to reference standards.

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 08-12-21. 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 initial calibration on 08-12-21. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-06-21, 10-07-21 and 11-01-21. A total of twelve continuing calibration standards were analyzed with analysis and re-analysis of 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 in each 24-hour. It was within the assigned QC limit for each anion.

4.13.3. Quality Control Samples consisted of two method blanks and two sets of LCS/LCSD. No sample was designated to be spiked as MS/MSD. A third set of QC (MB and LCS/LCD) was analyzed with dilutions and/or re-analysis of samples for Fluoride, Chloride and Sulfate 11-01-21. Recoveries of LCS/LCSD were all within 90-110 % of spiked values for each anion. Percent RPDs were less than 20% for LCS/LCSD.

4.13.4. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this sample delivery group.

4.13.5. Raw data was submitted for all requested field samples. Sample MW01102021 was designated to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met. Due to high concentrations of Sulfate and Chloride, samples were analyzed with high dilution factors on 11-01-21 within 28-day holding time.

5.0 CONCLUSION

SDG #21J038 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 #21J061 Analytical Data Package

Publication Date: 01-24-22

By
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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 10-06-21. EMAX Laboratories received the samples on 10-07-21. 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 TMW50102021(Lab ID# J061-01) was 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. 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 (9 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (8 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (7 samples)
EPA Method SW8332: Nitroglycerine and PETN (7 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) (6 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (5 samples)

EPA Method 6850: Perchlorate (8 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (8 samples)

EPA Method 7470A: Mercury & Dissolved Mercury (8 samples)
EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate (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 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 # 21J061 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 nine (9) water samples were collected on 10-06-21. EMAX Laboratories received the samples on 10-07-21.

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 TMW50102021 (EMAX ID #J061-01) from this sample delivery group was selected for stage 3 data review. 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; only for total metals
- 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#21J061 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW50102021	21J061-01	10-06-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC; 9056A
MW20102021	21J061-02	10-06-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH as Gasoline & as DRO Anions by IC; 9056A
QC06102021TB3	21J061-03	10-06-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW30102021	21J061-04	10-06-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; 9056A
BGMW09102021	21J061-05	10-06-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 Anions by IC; 9056A Chlorinated Herbicides Polychlorinated Biphenyls

Site Name: Fort Wingate, New Mexico

SDG#21J061 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW35102021	J061-06	10-06-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Anions by IC; 9056A
QC06102021EB2	J061-07	10-06-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC; 9056A
TMW37102021	J061-08	10-06-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; 9056A
TMW63102021	J061-09	10-06-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC; 9056A

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	SW8332	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	0.5µg/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	None. Holding times were met
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 and Dissolved Mercury	Water	Collection to Analysis: 28 days	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 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 1.7˚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:

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

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) =9/9X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =8/8X100=100%
Completeness (EPA Method 8330B: Explosives) =7/7X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =7/7X100=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) 6/6X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =5/5X100=100%
Completeness (EPA Method 6850: Perchlorate) =8/8X100=100%
Completeness (EPA Method 7470A: Dissolved and Total Mercury) =8/8X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =8/8X100=100%
Completeness (EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate) =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
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor
EPA **Method 9056A** for Anions 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. Nine water samples were collected on 10-06-21. Samples were analyzed on 10-13-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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 4.1.7.

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 10-07-21 and at the beginning of analysis shift on 10-13-21. 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 10-07-21. 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 10-07-21
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.

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

Target Analytes	Quadratic Regression 10-07-21
2-Chloro-1,1,1-trifluoroethane	0.9999

Average response factors for all target compounds were within method's recommended values, except for: Acetone (0.041), 2-Butanone (0.039) and 2-Hexanone (0.074). However, recoveries were all 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 10-07-21
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 10-08-21. 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 10-13-21. Prior to each continuing calibration, **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 10-13-21 (I)	Continuing cal. Response factors 10-13-21 (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 (10-13-21) I	%Deviation from Initial calibration (10-13-21) 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 the initial calibration was less than 20 percent for the rest of target list, except for the compounds listed in the table below.

Target compunds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-13-21) I	%Deviation from Initial calibration (10-13-21) II
2-Chloroethyl vinyl ether	≤ 20%	24.0%*	27.1%*
Tetrachlorohydrofuran	≤ 20%	--	24.2%*
2-Chloro-1,1,1-trifluoroethane	≤ 20%	--	99%*

*Outside QC acceptance limits

This should not affect the data quality.

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, except for traces of Methylene chloride at less than ½ LOQ. This compound was not detected in any of the field samples. 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 assigned to this sample delivery group.

4.1.7. Raw data was submitted for all samples. Sample TMW50102021 (Lab ID# 21J061-01) 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. Traces of Bromodichloromethane, Chloroform, Bromoform and Dibromochloromethane was detected in equipment blank. However, these compounds were not detected in any of the field samples.

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

4.2.1. Technical Holding Times

Holding time requirement was met for all samples. Eight water samples were collected on 10-06-21, extracted on 10-13-21 and were analyzed on 10-18-21 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 08-12-21, 08-13-21 and at the beginning of analysis shift on 10-18-21. 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 08-12-21 and 08-13-21. 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 08-12-21
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 08-12-21
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) 08-12-21**
Benzoic acid	0.9991
2,4-Dinitrophenol	0.9993
4-Nitrophenol	1.00
4,6-Dinitro-2-Methylphenol	0.9983
Benzidine	0.9994
3,3-dimethylbenzidine	0.9999

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 08-12-21 and 08-13-21. 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 except 4,6-Dinitro-2-methylphenol (25.0%). Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-13-21. 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 (10-18-21) 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 nearly 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 (10-18-21) 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 in both opening and closing daily standards.

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. Results and recoveries of LCS/LCSD was used to evaluate both accuracy and precision for this method. The full list of target compounds was spiked and reported for LCS/LCSD. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits for the entire compound list in LCS/LCSD. However, few compounds failed the maximum 20%RPD as listed in the table below:

Analytes	**Calculated** RPD%	**Maximum RPD%**
2,4-Dichlorophenol	22.0*	≤ 20
2-Chlorophenol	27.0*	≤ 20
2-Nitrophenol	23.0*	≤ 20
Benzoic acid	34.0*	≤ 20
Phenol	25.0*	≤ 20
2,6-Dichlorophenol	25.0*	≤ 20

*Exceeded acceptable maximum %RPD

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 p-Terphenyl in sample TMW50102021, as shown in the table below.

Surrogate	TMW50102021 (Lab ID#21J061-01)	**QC Limit%**
p-Terphenyl	47*%	50-134

*Exceeded lower acceptable limit

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 TMW50102021(Lab ID#21J061-01) 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 eight water samples requested for this method. Water samples were collected on 10-06-21, extracted on 10-12-21 and analyzed on 10-30-21 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 08-23-21 and before sample analysis on 10-30-21. 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 08-23-21. 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 08-14-21 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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 08-14-21 and 08-24-21. 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 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 10-30-21, 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, %D for Delta BHC failed the maximum 20% difference in the last two closing daily standards (24% and 31%). 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 one 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 except for one compound shown in the table below:

Surrogate	LCS	LCSD	**QC Limit%**
Endosulfan sulfate	149*%	143*%	62-133

* Exceeded acceptable QC limits

This should not affect the quality of data; LCS/LCSD recoveries were biased high and this compound was not detected in any of the field samples.

Raw data for the QC samples were matching the reported values.

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 assigned to this sample delivery group.

4.3.6. Raw data was submitted for all samples. Sample TMW50102021(Lab ID#21J061-01) 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.

4.4. 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 four water samples requested for this method. Water samples were collected on 10-06-21, extracted on 10-12-21, and analyzed on 10-16-21.

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 10-14-21. 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 10-14-21. 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 were injected at single point for identification only.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-15-21 and 10-16-21, 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 and its associated sample: No field duplicate sample was assigned to this sample delivery group.

4.4.6. Raw data was submitted for all samples. Sample TMW50102021(Lab ID#21J061-01) 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 four water samples requested for this method. Water samples were collected on 10-06-21, extracted on 10-13-21 and analyzed on 11-05-21.

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 10-28-21. 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.

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 10-28-21. 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 11-05-21, 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 compounds from both channels. One compound (Dicamba 24%) exceeded 20% limit in column B.

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 sample delivery group.

4.5.6. Raw data was submitted for all samples. Sample TMW50102021 (Lab ID#21J061-01) 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 seven (7) water samples requested for this method. Water samples were collected on 10-06-21, extracted on 10-12-21 and analyzed on 10-13-21 and 10-14-21, 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 08-23-21 and 08-24-21 for both primary and confirmation (Biphenyl) columns. 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 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 on 08-23-21 and 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% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of five continuing calibration standards were analyzed on 10-13-21and 10-14-21, bracketing the analyses of samples and all the QC samples. Daily standards for Biphenyl column were analyzed on 10-19-21 and 10-21-21. 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 only. No sample was designated to be spiked as MS/MSD. The full 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 assigned to this sample delivery group.

4.6.6. Raw data was submitted for all samples. Sample TMW50102021(Lab ID#21J061-01) was selected to be reviewed as stage 3 data deliverable. Raw data of 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. All positive results, if any were confirmed with confirmation 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 seven (7) water samples requested for this method. Water samples were collected on 10-06-21, extracted on 10-12-21 and analyzed on 10-13-21.

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 10-13-21, 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 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 within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** No field duplicate sample was assigned to this sample delivery group.

4.7.6. Raw data was submitted for all samples. Sample TMW50102021(Lab ID#21J061-01) was selected to be reviewed as stage 3 data deliverable. Raw data of 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. 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 six water samples requested for this method. water samples were collected on 10-06-21. Samples were analyzed on 10-08-21 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 09-23-21. 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 09-23-21. 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 10-08-21, 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 to this sample delivery group.

4.8.6. Raw data was submitted for all samples. Sample TMW50102021(Lab ID#21J061-01) 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 five water samples requested for this method. Water samples were collected on 10-06-21, extracted on 10-11-21 and analyzed on 10-12-21 and 10-13-21.

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 08-12-21. 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 08-12-21. 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 10-12-21 and 10-13-21 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 sample delivery group.

4.9.6. Raw data was submitted for all samples. Sample TMW50102021(Lab ID#21J061-01) 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 eight samples requested for this method. Water samples were collected on 10-06-21. Samples were analyzed on 10-12-21 and 10-13-21.

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 09-21-21. 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 0.999 (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 09-21-21. 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 10-12-21 and 10-13-21, 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. Perchlorate was spiked and reported for each set of LCS/LCSD. Percent recoveries (%R) were within the established QC limits 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 assigned to this sample delivery group.

4.10.6. Raw data was submitted for all samples. Sample TMW50102021 (Lab ID#21J061-01) 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. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.11.1. 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 10-06-21. Samples were prepared (digested) for both total and dissolved metals on 10-13-21. Samples and QC samples were analyzed on 11-02-21 and 11-05-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Two sets of initial calibration were established at the start of each analysis day on 11-02-21 and 11-05-21. Tune performance report was generated at the start of each analysis day, before initial calibration. It was within method’s acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard was used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

Metals	Concentration µg/L
Se, Pb, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Mn, Ni, Ag, Tl, V, and Zn	50,100,500,1000
Al, Fe, Ca, Mg, Na, and K	50,000

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of ±20% of the true value for the analytes incorporated in each solution.

4.11.3. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW50102021 was spiked as MS/MSD for total metals only. Recoveries of LCS/LCSD were all within the acceptance limit of 80-120% for total and dissolved metals. Recoveries of MS/MSD were mostly acceptable except the few metals as listed in the table below.

ANALYTE	Total Metals			Dissolved Metals		
	TMW50102021 MS%	TMW50102021 MSD%	**QC Limit%**	NA	NA	**QC Limit%**
Aluminum	120*	184*	84-117	NA	NA	84-117
Antimony	√	√	85-117	NA	NA	85-117
Arsenic	√	√	84-116	NA	NA	84-116
Barium	135*	284*	86-114	NA	NA	86-114
Beryllium	√	√	83-121	NA	NA	83-121
Cadmium	85*	85*	84-115	NA	NA	87-115
Calcium	√	√	87-118	NA	NA	87-118
Chromium	√	√	85-116	NA	NA	85-116
Cobalt	√	√	86-115	NA	NA	86-115
Copper	√	√	85-118	NA	NA	85-118
Iron	109	126*	87-118	NA	NA	87-118
Lead	√	√	88-115	NA	NA	88-115
Magnesium	109	135*	83-118	NA	NA	83-118
Manganese	120*	120*	87-115	NA	NA	87-115
Nickel	√	√	85-117	NA	NA	85-117
Potassium	√	√	85-115	NA	NA	85-115
Selenium	√	√	80-120	NA	NA	80-120
Silver	√	√	85-116	NA	NA	85-116
Sodium	167*	267*	85-117	NA	NA	85-117
Thallium	√	√	82-116	NA	NA	82-116
Vanadium	√	√	86-115	NA	NA	86-115
Zinc	√	√	83-119	NA	NA	83-119

*Outside control limits

As a result, all the positive results for the parent sample will be qualified as estimated value “J” and non-detect results as “UJ” for these metals. This was attributed to high dilution factor and matrix interference of the parent sample in the case narrative.

Method blank results for metals were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard. Sample TMW50102021 was used for serial dilution for total metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis except for Manganese (11%) and Vanadium (12%). The same sample was used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total metals.

4.11.4. Field duplicate sample and its associated sample: No field duplicate was assigned to this sample delivery group.

4.11.5. Raw data was submitted for all samples. Sample TMW50102021 (Lab ID#21J061-01) was selected to be reviewed as stage 3 data deliverable. Raw data of 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.

4.12. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.12.1. 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 10-06-21. Samples were prepared (digested) and analyzed for Mercury and dissolved Mercury on 10-18-21. All samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 10-18-21. The correlation coefficient of at least 0.999980 was calculated to show the linearity of calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

Initial calibration verification and Continuing calibration verification standards for mercury was within the acceptable range (90-110% of the spiked value).

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD for Mercury and dissolved Mercury. Recoveries of LCS/LCSD and were within acceptable range of 82-119%. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this sample delivery group.

4.12.5. Raw data was submitted for all samples. Samples TMW50102021(Lab ID#21J061-01) 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 raw data, agreed with all the results reported in data summary reports.

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

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of eight water samples requested for this method. Water samples were collected on 10-06-21. Samples were analyzed on 10-07-21 and 10-08-21 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Some samples were re-analyzed for Bromide, Chloride, Fluoride and Sulfate within 28-day holding time on 11-02-21.

4.13.2. 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 on the basis of retention time as compared to reference standards.

The instruments were initially calibrated with nine calibration levels (0.05-20mg/L) on

08-12-21 and 10-21-21. 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 component. 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 initial calibration on 08-12-21 and 10-21-21. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-07-21, 10-08-21 and 11-02-21. A total of twelve continuing calibration standards were analyzed with re-analysis of some 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 in each 24-hour. It was within the assigned QC limit for each anion.

4.13.3. Quality Control Samples consisted of two method blanks and two sets of LCS/LCSD. No sample was designated to be spiked as MS/MSD. A third set of QC (MB and LCS/LCD) was analyzed with dilutions and /or re-analysis of samples for Bromide, Chloride, Fluoride and Sulfate with a second instrument on 11-02-21. Recoveries of LCS/LCSD were all within 90-110 % of spiked values for each anion. Percent RPDs were less than 20% for LCS/LCSD.

4.13.4. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this sample delivery group.

4.13.5. Raw data was submitted for all requested field samples. Sample TMW50102021 was selected to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met. Due to high concentrations of Sulfate and Chloride, samples were analyzed with high dilution factors on 11-02-21 within 28-day holding time.

5.0 CONCLUSION

SDG #21J061 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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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #21J071 Analytical Data Package

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 10-07-21. EMAX Laboratories received the samples on 10-08-21. 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 BGMW12102021 (Lab ID#J071-05) 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. Sample TMW15102021 (Lab ID#J071-07) was designated to be spiked as MS/MSD. Raw data for this sample together with 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 (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 initial and re-analysis of all samples. However, sample TMW15102021(Lab ID#J071-07) was analyzed for Nitrate, Nitrite and Orthophosphate five hours past 48-hour holding time due to standard failure. This sample was later re-sampled on 10-15-21 and was reported with a new sample delivery group (SDG#21J146). The deviations, are discussed in Section 4.0 for this method.

The SDG # 21J071 analytical data evaluated in this data validation report 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 10-07-21. EMAX Laboratories received the samples on 10-08-21.

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 BGMW12102021 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. Sample TMW15102021 was designated to be spiked as MS/MSD on the chain of custody. Raw data for this sample together with other QC samples was also 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 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;
- LCSs 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#21J071 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
TMW31S102021	J071-01	10-07-21	S3VM	Anions by IC,
BGMW10102021	J071-02	10-07-21	S3VM	Anions by IC,
BGMW10102021D	J071-03	10-07-21	S3VM	Anions by IC,
TMW10102021	J071-04	10-07-21	S3VM	Anions by IC,
BGMW12102021	J071-05	10-07-21	S3VM	Anions by IC,
TMW08102021	J071-06	10-07-21	S3VM	Anions by IC,
TMW15102021	J071-07	10-07-21	S3VM	Anions by IC,
TMW15102021MS	J071-07M	10-07-21	S3VM	Anions by IC,
TMW15102021MSD	J071-07S	10-07-21	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 one sample*
		Analysis within 28 days for Bromide, Fluoride, Chloride and Sulfate	None. Holding times were met

*One sample, TMW15102021 was analyzed five hours past 48-hour holding time for Nitrite, Nitrate and Orthophosphate. This sample was later re-sampled on 10-15-21 and was reported with a new sample delivery group (SDG#21J146).

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 4.9˚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 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:

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

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 10-07-21. Samples were analyzed on 10-08-21 and 10-09-21 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Some samples for Bromide, Chloride, fluoride and Sulfate were analyzed and re-analyzed with dilution on 11-02-21 and 11-03-21, within 28-day holding time. Analysis was within 28-day holding time for Bromide, Chloride, Fluoride and Sulfate. One sample (TMW15102021) was analyzed for Nitrate, Nitrite and Orthophosphate five hours past 48-hour holding time. This sample was re-sampled on 10-15-21 and the results were reported with a new sample delivery group (SDG#21J146).

4.1.2. Initial and continuing calibration: Anions such as Bromide, Chloride, Fluoride, 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 on the basis of retention time as compared to reference standards.

Two instruments were initially calibrated with nine calibration levels (0.05-20mg/L) on 08-12-21 and 10-21-21. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout the analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. 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 08-12-21 and 10-21-21. Recoveries were all within 90-110% of expected true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-08-21 10-09-21, 11-02-21 and 11-03-21. A total of four continuing calibration standards were initially analyzed on 10-08-21 and 10-09-21. Later, six more continuing calibration standards were analyzed on 11-02-21 and 11-03-21. 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.

4.1.3. Quality Control Samples consisted of one method blank and one set of LCS/LCSD, MS/MSD and sample/sample duplicate for initial analysis of all anions. Sample TMMW15102021was designated to be spiked as MS/MSD and sample/sample duplicate analysis. Two more sets of quality control samples (MB and LCS/LCD) were also analyzed on 11-02-21 and 11-03-21 with re-analysis and /or dilution of samples for Bromide, Chloride, Fluoride and Sulfate. Recoveries of LCS/LCSDs were all within 90-110 % of spiked values for each anion. Recoveries of MS/MSD were mostly within acceptable limits except for two anions as indicated in the table below:

Anion	TMW15102021 MS%	TMW15102021 MSD%	% Acceptance limits
Orthophosphate	62*	64*	80-116
Bromide	68*	68*	91-110

*Outside Acceptance limits

Therefore, the reported results will be qualified as estimated value (“J”) for positive results and as (“UJ”) for non-detect results in the parent sample for these anions. Percent RPDs were less than 20% for LCS/LCSD and MS/MSD. Sample TMW15102021 was also used for sample/sample duplicate analysis. All the recoveries and % RPDs were within acceptance limits for each anion.

4.1.4. Field duplicate sample and its associated sample: Sample BGMW10102021 was identified as field duplicate of BGMW10102021D. Results for sample/sample duplicate is summarized in the table below:

Anion	BGMW10102021 (Lab ID #J071-02) mg/L	BGMW10102021D (Lab ID #J071-03) mg/L	**% RPD**
Nitrate	0.065J	0.072J	10.2
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.36J	0.36J	<1
Chloride	70	70	<1
Fluoride	0.56	0.56	<1
Sulfate	780	800	2.53

4.1.5. Raw data was submitted for all requested field samples. Sample BGMW12102021 was selected to be reviewed as stage 3 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. Samples were analyzed and/or re-analyzed for Bromide, Fluoride, Sulfate and Chloride, on 11-02-21 and 11-03-21 within 28-day holding time. As mentioned in section 4.1.1, one sample (TMW15102021) was analyzed for Nitrate, Nitrite and Orthophosphate five hours past 48-hour holding time. This sample was re-sampled on 10-15-21 and the results were reported with a new sample delivery group (SDG#21J146). Therefore, no qualification will be applied to this sample.

5.0 CONCLUSION

SDG #21J071 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 #21J089 Analytical Data Package

Publication Date: 01-25-22

By
ZIBA HOSSEINI

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TABLE OF CONTENTS

EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 10-08-21. EMAX Laboratories received the samples on 10-09-21. 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. Four field samples; TMW41102021 (EMAX ID #J089-06), TMW39S102021 (EMAX ID #J089-07), TMW22102021 (EMAX ID #J089-08) and TMW55102021 (EMAX ID #J089-09) were designated as stage 3 deliverable on the chain of custody. Raw data for these samples was compared to the reported summary tables for each method and went through comprehensive data validation. Sample TMW01102021(Lab ID# J089-05) was designated to be spiked as MS/MSD on the chain of custody. Raw data for this sample together with method blank and LCS/LCSD for each method 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, (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 (11 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (15 samples)
EPA Method SW8332: Nitroglycerine and PETN (15 samples)
EPA Method 8081B: Organochlorine Pesticides (8 samples)
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 (14 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (16 samples)

EPA Method 7470A: Mercury & Dissolved Mercury (16 samples)

EPA Method SW9056A: Anions by IC (16 samples)

1,4-Dioxane by 8270 SIM: (2)

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 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. However, at least eleven samples failed the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate analysis. These samples were later re-sampled and reported with a new sample delivery group. The deviations, if any, are discussed in Section 4.0 for each method.

The SDG # 21J089 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. Otherwise, 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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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.

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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 seventeen (17) water samples were collected on 10-08-21. EMAX Laboratories received the samples on 10-09-21.

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. Four field samples; TMW41102021 (EMAX ID #J089-06), TMW39S102021 (EMAX ID #J089-07), TMW22102021 (EMAX ID #J089-08) and TMW55102021 (EMAX ID #J089-09) from this sample delivery group were designated for stage 3 data review. Raw data for these samples was evaluated comprehensively. Sample TMW01102021 (EMAX ID #J089-05) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with other QC samples in this report LCS/LCSD was also 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#21J089 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW23102021	21J089-01	10-08-21	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; Method 9056A
TMW07102021	21J089-02	10-08-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline; TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Anions by IC; Method 9056A
QC08102021TB5	21J089-03	10-08-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW33102021	21J089-04	10-08-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Anions by IC; Method 9056A
TMW01102021	21J089-05	10-08-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; Method 9056A
TMW41102021	21J089-06	10-08-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury& Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; Method 9056A

Site Name: Fort Wingate, New Mexico

SDG#21J089 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW39S102021	J089-07	10-08-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; Method 9056A
TMW22102021	J089-08	10-08-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC; Method 9056A
TMW55102021	J089-09	10-08-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC; Method 9056A
TMW29102021	J089-10	10-08-21		VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; Method 9056A
TMW02102021	J089-11	10-08-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; Method 9056A

Site Name: Fort Wingate, New Mexico

SDG#21J089 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW02102021D	J089-12	10-08-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; Method 9056A
MW27102021	J089-13	10-08-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Anions by IC; Method 9056A 1,4-Dioxane by 8270SIM
TMW62102021	J089-14	10-08-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Anions by IC; Method 9056A
SMW01102021	J089-15	10-08-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; Method 9056A

Site Name: Fort Wingate, New Mexico

SDG#21J089 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW30102021	J089-16	10-08-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Anions by IC; Method 9056A
QC08102021EB3	J089-17	10-08-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Anions by IC; Method 9056A 1,4-Dioxane by 8270SIM
TMW01102021MS	J089-05M	10-08-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; Method 9056A
TMW01102021MSD	J089-05S	10-08-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC; Method 9056A

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
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	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	0.5µg/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-two 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	None. Holding times were met
Anions by IC	water	Analysis 48 hours from collection For Nitrate, Nitrite and Orthophosphate; 28 days from collection for the rest	Holding times were met for all except for eleven samples for Nitrate and Nitrite*; and one sample for Orthophosphate**
Mercury and Dissolved Mercury	Water	Collection to Analysis: 28 days	None. Holding times were met

Samples: TMW23102021*, TMW07102021*, TMW33102021*, TMW01102021*, TMW41102021*, TMW39S102021*, TMW22102021*, TMW55102021*, TMW02102021*, TMW02102021D*, SMW01102021*& SMW01102021**; All re-sampled and analyzed with a new sample delivery group (21J146)

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-two ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler’s temperature was reported as low as 1.0˚C and as high as 4.7˚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) =17/17X100=100%

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

Completeness (EPA Method 8330B: Explosives) =15/15X100=100%

Completeness (EPA Method 8332: Nitroglycerine & PETN) =15/15X100=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) =14/14X100=100%

Completeness (EPA Method 7470A: Mercury and Dissolved Mercury) =16/16X100=100%

Completeness (EPA Method 6020A: Dissolved and Total Metals) =16/16X100=100%

Completeness (EPA Method SW9056A: Anions) =16/16X100=100%

Completeness (EPA Method 3520B/8270SIM: 1,4-Dioxane) =2/2X100=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
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury and Dissolved Mercury by Cold Vapor
EPA **Method 9056A** for Nitrate, Nitrite, Orthophosphate, Bromide, Fluoride, Chloride and Sulfate by IC
EPA Method **3520C/8270SIM** for 1,4-Dioxane by GC/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 seventeen samples. Water samples were collected on 10-08-21. Samples were analyzed on 10-13-21, 10-14-21 and 10-18-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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 4.1.7.

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 10-11-21 and at the beginning of each analysis shift on 10-13-21, 10-14-21 and 10-18-21. 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 10-11-21. 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 10-11-21
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.

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

Target Analytes	**Least Square Linear Regression (CCF) 10-11-21**
Vinyl acetate	0.9982

Average response factors for all target compounds were within method's recommended values, except for: Acetone (0.084). 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 10-11-21**
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 10-12-21. 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, except for Vinyl acetate (30.2%).

Continuing calibration check standards were analyzed at the beginning and end of each analysis shift on 10-13-21, 10-14-21 and 10-18-21. Prior to each continuing calibration,

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 10-13-21 (I& II)	Continuing cal. Response factors 10-14-21 (I& II)	Continuing cal. Response factors 10-18-21 (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 (10-13-21) I&II	%Deviation from Initial calibration (10-14-21) I&II	%Deviation from Initial calibration (10-18-21) 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

In all continuing calibration verification standards analyzed, deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds listed in the table below for each standard. This should not affect data quality.

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-13-21)		%Deviation from Initial calibration (10-14-21)		%Deviation from Initial calibration (10-18-21)	
		opening	closing	opening	closing	opening	closing
Methyl aetate	≤ 20%	√	√	√	29.4	√	√
Methylene chloride	≤ 20%	√	21.3%	√	√	√	√
4-Methyl-2-pentanone	≤ 20%	√	38.0%	√	26.7%	√	√
2-Hexanone	≤ 20%	√	22.3%	√	21.4%	√	√
Dichlorofluoromethane	≤ 20%	√	√	√	25.1%	√	26.5%
2-Butanone	≤ 20%	√	√	√	20.7%	√	√
2-Cloroethyl vinyl ether	≤ 20%	√	√	√	√	25.9%	21.2%
Hexachlorobutadiene	≤ 20%	√	√	√	√	√	√

4.1.5. Quality Control samples reported consisted of three method blanks, three sets of LCS/LCSD and MS/MSD. Sample TMW01102021 (lab ID #J089-05) was designated to be analyzes as MS/MSD.   The full list of target compounds was spiked and reported for MS/MSD and 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, except for traces of Methylene chloride at less than ½ LOQ. This compound was not detected in any of the field samples.

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

4.1.6. **Field duplicate sample** and its associated sample: Sample TMW02102021 was identified as field duplicate of TMW02102021D. Both sample and corresponding field duplicate sample were reported as non-detected for all volatile compound list.

4.1.7. Raw data was submitted for all samples. Four samples: TMW4102021, TMW39S102021, TMW22102021 and TMW55102021 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. Traces of Bromodichloromethane, Chloroform, Bromoform and Dibromochloromethane was detected in equipment blank. However, these compounds were not detected in any of the field samples.

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

4.2.1. Technical Holding Times

Holding time requirement was met for all samples. Eleven (11) water samples were collected on 10-08-21, extracted on 10-15-21 and were analyzed on 10-19-21 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 08-12-21, 08-13-21 and at the beginning of analysis shift on 10-19-21. 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 08-12-21 and 08-13-21. 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 08-12-21
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 08-12-21
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. All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

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) 08-12-21**
Benzoic acid	0.9991
2,4-Dinitrophenol	0.9993
4-Nitrophenol	1.00
4,6-Dinitro-2-Methylphenol	0.9983
Benzidine	0.9994
3,3-dimethylbenzidine	0.9999

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 08-12-21 and 08-13-21. 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 except 4,6-Dinitro-2-methylphenol (25.0%). Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-19-21. 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 (10-19-21) 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 nearly 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 (10-19-21) 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 initial calibration was less than 20 percent for the rest of target list except for the compounds listed below in each continuing calibration standard:

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (10-19-21) I	%Deviation from Initial calibration (10-19-21) II
3,3-Dimethylbenzidine	≤ 20%	23.4%*	--
2-Nirtoaniline	≤ 20%	--	23.8%*
Hexachlorocyclopentadiene	≤ 20%	--	27.3%*

*Exceeded 20% maximum allowed difference in the opening/closing daily standards

This deviation should not affect quality of the presented data.

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 for all the compounds reported in the QC samples were within the project acceptance limits for the entire compound list in LCS/LCSD. However quite a number of compounds (17) exceeded maximum 20% RPD limit. Recoveries of LCS/LCSD was used to evaluate accuracy and precision for 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 sample analysis was requested for this method.

4.2.7. Raw data was submitted for all samples. Four samples; TMW4102021, TMW39S102021, TMW22102021 and TMW55102021 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.

4.3. 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 water samples requested for this method. Eight (8) water samples were collected on 10-08-21, extracted on 10-15-21 and analyzed on 10-26-21 and 10-27-21 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 08-23-21 and before sample analysis on 10-26-21 and 10-27-21. 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 08-23-21. 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 08-14-21 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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 08-14-21 and 08-24-21. 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 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 10-26-21 and 10-27-21, 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 one 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 all within established QC limits. Raw data for the QC samples were matching the reported values.
Results for method blank were reviewed 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 assigned to this method.

4.3.6. Raw data was submitted for all samples. Three samples; TMW4102021, TMW39S102021, and TMW55102021 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.

4.4. 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 five water samples requested for this method. Water samples were collected on 10-08-21, extracted on 10-15-21, and analyzed on 10-19-21, 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.

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 10-14-21. 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 10-14-21. 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 would be injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-19-21, 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 channels.

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 collected for this method.

4.4.6. Raw data was submitted for all samples. Sample TMW55102021 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.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for five water samples requested for this method. Water samples were collected on 10-08-21, extracted on 10-15-21 and analyzed on 10-29-21.

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 10-28-21. 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.

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 10-28-21. 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 10-29-21, 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 compounds from channels A and B.

Results for surrogate recoveries and QC were all reported from channel B. Channel A 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 for this method.

4.5.6. Raw data was submitted for all samples. Sample TMW55102021 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.

4.6. 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 10-08-21, extracted on 10-14-21 and analyzed on 10-14-21, 10-15-21 and 10-18-21, 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 08-24-21. Confirmation column (Kinetex-Biphenyl) 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 08-24-21. 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 eight continuing calibration standards (both primary and confirmation columns) were analyzed on 10-14-21, 10-15-21 and 10-18-21, 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 and MS/MSD. Sample TMW01102021 was designated to be spiked as MS/MSD. The full explosive target list was 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 TMW02102021 was identified as field duplicate of sample TMW02102021D. Traces of 3,5-Dinitroaniline was detected in each sample as listed in the table below:

Analyte	TMW02102021 µg/L	TMW02102021D µg/L	% RPD
3,5-Dinitroaniline	0.31J	0.20J	43.1

4.6.6. Raw data was submitted for all samples. Four samples; TMW4102021, TMW39S102021, TMW22102021 and TMW55102021 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 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 10-08-21, extracted on 10-14-21 and analyzed on 10-15-21 within the required holding time.

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 10-15-21, 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 TMW01102021 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 within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Sample TMW02102021 was identified as field duplicate of sample TMW02102021D. No explosive target compound was detected in either sample or field duplicate sample.

4.7.6. Raw data was submitted for all samples. Four samples; TMW4102021, TMW39S102021, TMW22102021 and TMW55102021 were designated to be reviewed as stage 3 data deliverable. Raw data of 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.

4.8. 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 10-08-21. Samples were analyzed on 10-14-21 and 10-15-21 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 09-23-21. 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 09-23-21. 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 10-14-21 and 10-15-21, 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 collected for this method.

4.8.6. Raw data was submitted for all samples. Sample TMW55102021 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 samples requested for this method. Water samples were collected on 10-08-21, extracted on 10-14-21 and analyzed on 10-15-21.

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 08-12-21. 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 08-12-21. 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 10-15-21 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 collected for this method.

4.9.6. Raw data was submitted for all samples. Sample TMW55102021 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 samples requested for this method. Water samples were collected on 10-08-21. Samples were analyzed on 10-14-21 and 10-15-21, 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 09-21-21. 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 0.999 (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 09-21-21. 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 10-14-21 and 10-15-21, 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 TMW01102021 was designated to be spiked as MS/MSD. Perchlorate 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 blanks was reviewed for each compound and no target was found in the method blanks. No surrogate is used in this method.

4.10.5. Field duplicate sample and its associated sample: Sample TMW02102021 was identified as field duplicate of TMW02102021D. Results for sample/sample duplicate is summarized in the table below:

Analyte	TMW02102021 µg/L	TMW02102021D µg/L	**% RPD**
Perchlorate	6.4	6.1	4.8

4.10.6. Raw data was submitted for all samples. Two samples TMW22102021 and TMW55102021 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. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sixteen water samples requested for this method. Water samples were collected on 10-08-21. Samples were prepared (digested) for both total and dissolved metals on 10-15-21. Samples and QC samples were analyzed on 11-08-21 and 11-09-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Two sets of initial calibration was performed at the start of each analysis day on 11-08-21 and 11-09-21. Tune performance report was generated at the start of each analysis day, before initial calibration. It was within method's acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. The correlation coefficient of at least 0.999 was calculated to show the linearity of calibration curve for each metal. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

Metals	Concentration µg/L
Se, Pb, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Mn, Ni, Ag, Tl, V, and Zn	50,100,500,1000
Al, Fe, Ca, Mg, Na, and K	50,000

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of ±20% of the true value for the analytes incorporated in each solution.

4.11.3. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW01102021 was designated to be spiked as MS/MSD. Recoveries of LCS/LCSD were all within the acceptance limit of 80-120% for both total and dissolved metals. Recoveries of MS/MSD were mostly acceptable except the few metals as listed in the table below. As a result, all the positive results will be qualified as estimated value “J” and non-detect results as “UJ” in the parent sample. This was attributed to high dilution factor and/or matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	TMW01102021 MS%	TMW01102021 MSD%	**QC Limit%**	TMW01102021 MS%	TMW01102021 MSD%	**QC Limit%**
Aluminum	√	√	84-117	√	√	84-117
Antimony	√	√	85-117	√	√	85-117
Arsenic	√	√	84-116	√	√	84-116
Barium	√	√	86-114	√	√	86-114
Beryllium	√	√	83-121	√	√	83-121
Cadmium	85*	86*	87-115	83*	84	87-115
Calcium	133*	67*	87-118	0*	67*	87-118
Chromium	65*	66*	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	√	√	85-118
Iron	√	√	87-118	√	√	87-118
Lead	√	√	88-115	√	√	88-115
Magnesium	√	√	83-118	77*	93	83-118
Manganese	√	√	87-115	86*	88	87-115
Nickel	78*	80*	85-117	√	√	85-117
Potassium	√	√	85-115	√	√	85-115
Selenium	√	√	80-120	√	√	80-120
Silver	√	√	85-116	√	√	85-116
Sodium	533*	567*	85-117	-367*	-433*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

Method blank results for metals were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard. Sample TMW01102021 was used for serial dilution for both total and dissolved metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis. The same sample was used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for both total and dissolved metals.

4.11.4. Field duplicate sample and its associated sample: Sample TMW02102021 was identified as field duplicate of TMW02102021D. Results for sample/sample duplicate is summarized in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW02102021 µg/L	TMW02102021D µg/L	% RPD	TMW02102021 µg/L	TMW02102021D µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	1.3J	1.4J	7.41	1.1J	1.1J	<1
Barium	7.4	7.1	4.14	6.9	6.7	2.94
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	24000	24000	<1	23000	23000	<1
Chromium	0.83J	U	200	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	U	U	--	U	U	--
Lead	U	U	--	U	U	--
Magnesium	3200	2900	9.84	2800	2800	<1
Manganese	U	U	--	U	U	--
Nickel	U	U	--	U	U	--
Potassium	1300	1300	<1	1200	1300	8.0
Selenium	78	76	2.60	75	75	<1
Silver	U	U	--	U	U	--
Sodium	1000000	1000000	<1	1000000	1000000	<1
Thallium	U	U	--	U	U	--
Vanadium	41	40	2.47	38	38	<1
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Samples TMW41102021, TMW39S102121, TMW22102021 and TMW55102021 were designated to be reviewed as stage 3 data deliverable. Raw data of 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.

4.12. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.12.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for sixteen (16) water samples requested for this method. Water samples were collected on

10-08-21. Samples were prepared (digested) and analyzed for Mercury and dissolved Mercury on 10-21-21. All samples were preserved and filtered for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 10-21-21. The correlation coefficient of at least 0.999453 was calculated to show the linearity of calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

Initial calibration verification and Continuing calibration verification standards for mercury were within the acceptable range (90-110% of the spiked value).

4.12.3. Quality Control: The QC samples consisted of method blank, LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Sample TMW01102021 was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW01102021 was also used for serial dilution. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample TMW02102021 was identified as field duplicate of TMW02102021D. No target compound was detected in sample and corresponding field duplicate sample for Mercury and dissolved Mercury.

4.12.5. Raw data was submitted for all samples. Samples TMW41102021, TMW39S102121, TMW22102021 and TMW55102021 were designated to be reviewed as stage 3 data deliverable. Raw data of 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.

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

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of sixteen water samples requested for this method. Water samples were collected on 10-08-21. Samples were analyzed on 10-08-21, 10-09-21, 10-10-21, 10-12-21 and 10-13-21 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. At least eleven water samples (TMW23102021, TMW0710202, TMW33102021, TMW01102021, TMW41102021, TMW39S102021, TMW 22102021, TMW55102021, TMW02102021, TMW02102021D and SMW01102021) failed 48-hour holding time for initial analysis of Nitrate and Nitrite. One sample (SMW01102021) failed 48-hour hooding time for Orthophosphate. Later, these samples were all re-sampled and analyzed with a new sample delivery group (SDG# 21J146).

Samples were analyzed and/or re-analyzed with dilutions for Bromide, Chloride, Fluoride and Sulfate on 11-03-21 and 11-04-21 within 28-day holding time.

4.13.2. Initial and continuing calibrations: Anions such as Bromide, Chloride, Fluoride, 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 on the basis of retention time as compared to reference standards.

Two instruments were initially calibrated with nine calibration levels (0.05-20mg/L) on 08-12-21 and 10-21-21. 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 component. 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 initial calibration on 08-12-21 and 10-21-21. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-08-21, 10-10-21, 10-12-21, 10-13-21, 11-03-21 and 11-04-21. A total of twenty-three continuing calibration standards were analyzed with analysis/re-analysis of samples. In all continuing calibration standards submitted, the recoveries of target anions were within

90-110% of the expected values. If one or more anions failed in a daily calibration standard, the associated samples were re-analyzed with reference to a new daily standard. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions.

4.13.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD, MS/MSD and sample/sample duplicate for initial analysis of all anions. Sample TMW01102021was designated to be spiked as MS/MSD and sample/sample duplicate analysis. Three more sets of MB and LCS/LCD were also analyzed with re-analysis and /or dilutions of samples for Nitrate, Nitrite, Bromide and Fluoride. Five sets of LCS/LCSD were analyzed with Chloride, and six sets of LCS/LCSD were analyzed with Sulfate. Recoveries of LCS/LCSDs were mostly within 90-110 % of spiked values for each anion. Recoveries of MS/MSD were within acceptable limits. Two anions, as indicated in the table below failed the QC limits.

Anion	TMW01102021 MS%	TMW01102021 MSD%	LCS%	LCSD%	% Acceptance limits
Orthophosphate	NA	NA	120*	121*	80-116
Bromide	68*	71*	NA	NA	91-110

*Outside Acceptance limits

Therefore, the reported results will be qualified as estimated value (“J”) for positive results and as (“UJ”) for non-detect results in the parent sample for Bromide. Percent RPDs were less than 20% for LCS/LCSD and MS/MSD. Sample TMW01102021 was also used for sample/sample duplicate analysis. All the recoveries and % RPDs were within acceptance limits for each anion.

4.13.4. Field duplicate sample: and its associated sample: Sample TMW02102021 was identified as field duplicate of TMW02102021D. Results for sample/sample duplicate is summarized in the table below:

Anion	TMW02102021 (Lab ID #J089-11) mg/L	TMW02102021D (Lab ID #J089-12) mg/L	**% RPD**
Nitrate	78	76	2.60
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	1.2	1.2	<1
Chloride	300	350	15.4
Fluoride	U	U	--
Sulfate	1400	1300	7.41

4.13.5. Raw data was submitted for all requested field samples. Four samples; TMW41042021, TMW39S102021, TMW22102021 and TMW55102021 were designated to be reviewed as stage 3 deliverable. Raw data for these samples together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met. As mentioned in section 4.13.1, eleven water samples failed 48-hour required holding time for Nitrate and Nitrite and one sample failed holding time (48-hour) for Orthophosphate. Later they were all re-sampled and analyzed for anions. Results were reported with a new sample delivery group (SDG#21J146).

4.14. 1,4-Dioxane (EPA Method 3520C/8270SIM)

4.14.1. Technical Holding Times

Holding time requirement was met for all samples. Two water samples were collected on 10-08-21, extracted on 10-15-21 and were analyzed on 10-18-21; within holding time.

Selective Ion Monitoring (SIM) method with GC/MS was used for analysis.

Isotopically-labeled 1,4-Dioxane-d8 was added to serve as internal standard.

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 4.14.7.

4.14.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 08-04-21 and at the beginning of analysis batch on 10-18-21. It passed all the method assigned criteria.

4.14.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 08-04-21. A multi-level calibration standard ranging from 0.05mg/L to 50mg/L was used for this purpose. Internal standard curve type in Selective Ion Monitoring (SIM) mode was used for initial calibration and all following analysis. 1,4-Dioxane-d8 was used as internal standard. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table. Average response curve type was used for 1,4-Dioxane and Bromobenzene as surrogate. Target compounds met the maximum 15% RSD limit (all less than 15%).

4.14.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 08-04-21. Percent difference (%D) were within ±20% of the expected value.

Continuing calibration check standard was analyzed at the beginning and at the end of analysis shift on 10-18-21. Prior to opening continuing calibration standard, instrument performance tune check standard (BFB) was carried out. It passed all the method tuning criteria. Percent difference (%D) and/or drift for both opening and closing continuing calibration standards were within ±20% of the expected value for each daily standard.

4.14.5. Quality Control: The QC samples reported consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. 1,4-Dioxane as the only target with Bromobenzene as surrogate was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits.

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 method's acceptance limit.

4.14.6. **Field duplicate sample** and its associated sample: No field duplicate sample was assigned for this method.

4.14.7. Sample MW27102021 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 #21J089 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. As mentioned in section 4.13.1, eleven samples failed 48-hour required holding time for Nitrate and Nitrite analysis, and one sample failed 48-hour holding time for Orthophosphate. Later, these sample were all re-sampled and analyzed within holding time with a new sample delivery group (SDG#21J146).

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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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 #21J090 Analytical Data Package

Publication Date: 01-25-22

By
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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 10-07-21. EMAX Laboratories received the samples on 10-09-21. 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 BGMW12102021(Lab ID# J090-06) 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. Sample TMW15102021(Lab ID#J090-08) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with method blank and LCS/LCSD for each method 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, (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 (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 (3 sample)
EPA Method 8151A: Chlorinated herbicides (3 sample)

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)
EPA Method SW6020A: Dissolved and total Metals by ICP-MS (7 samples)
EPA Method 7470A: Mercury & Dissolved Mercury (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 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 # 21J090 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 10-07-21. EMAX Laboratories received the samples on 10-09-21.

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 BGMW12102021 (EMAX ID #J090-06) from this sample delivery group was selected for stage 3 data review. Raw data for this sample was evaluated comprehensively. Sample TMW15102021 (EMAX ID #J090-08) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with other QC samples in this report LCS/LCSD was also 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#21J090 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW31S102021	21J090-01	10-07-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
QC07102021TB4	21J090-02	10-07-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
BGMW10102021	21J090-03	10-07-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850
BGMW10102021D	21J090-04	10-07-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850
TMW10102021	21J090-05	10-07-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#21J090 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
BGMW12102021	J090-06	10-07-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
TMW08102021	J090-07	10-07-21	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS TPH Gasoline; TPH as DRO Perchlorate by 6850
TMW15102021	J090-08	10-07-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850
TMW15102021MS	J090-08M	10-07-21		VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850
TMW15102021MSD	J090-08S	10-07-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS 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	SW8332	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,200 µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Perchlorate	SW6850	0.5µg/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 eleven 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	None. Holding times were met
Mercury and Dissolved Mercury	Water	Collection to Analysis: 28 days	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 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 eleven ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.0˚C and as high as 3.2˚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:

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

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) =8/8X100=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) =3/3X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =3/3X100=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 (EPA Method 7470A: Mercury and Dissolved Mercury) =7/7X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =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 **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 **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury and Dissolved Mercury by Cold Vapor

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 10-07-21. Samples were analyzed on 10-12-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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 4.1.7.

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 07-23-21 and at the beginning of analysis shift on 10-12-21. 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 07-23-21. 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 07-23-21
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.

Average response factors for all target compounds were within method’s recommended values, except for: Acetone (0.036) and 2-Butanone (0.065). 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 07-23-21
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 07-24-21. 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 10-12-21. Prior to continuing calibration analysis, **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 10-12-21 (I)	Continuing cal. Response factors 10-12-21 (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 (10-12-21) I	%Deviation from Initial calibration (10-12-21) 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 the initial calibration was less than 20 percent for the rest of target list, except for Chloromethane (20.9%) in the closing daily standard run on 10-12-21. This should not affect data quality.

4.1.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW15102021 (lab ID #J090-08) was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for MS/MSD and 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: Sample BGMW10102021 was identified as field duplicate of BGMW10102021D. Both sample and corresponding field duplicate sample were reported as non-detected for all volatile compound list.

4.1.7. Raw data was submitted for all samples. Sample BGMW12102021 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 five water samples. water samples were collected on 10-07-21, extracted on 10-14-21 and were analyzed on 10-18-21 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 08-17-21, 08-18-21 and at the beginning of analysis shift on 10-18-21. 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 08-17-21 and 08-18-21. 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 08-17-21
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 15%) 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 08-17-21
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	√

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 08-17-21
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) 08-17-21
Benzoic acid	0.9963
2,4-Dinitrophenol	0.9988

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

4.2.4. Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard on 08-17-21 and 08-18-21. 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 10-18-21. 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 (10-18-21) 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 (10-18-21) 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.

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 for the entire compound list in LCS/LCSD. Recoveries of LCS/LCSD was used to evaluate accuracy and precision for 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: Sample BGMW10102021 was identified as field duplicate of BGMW10102021D. Both sample and corresponding field duplicate sample were reported as non-detected for the full Semi-Volatile Compound list.

4.2.7. Raw data was submitted for all samples. Sample BGMW12102021 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 all water samples requested for this method. Five (5) water samples were collected on 10-07-21, extracted on 10-13-21 and analyzed on 10-28-21 and 10-29-21 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 08-23-21 and before sample analysis on 10-28-21 and 10-29-21. 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 08-23-21. 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 08-14-21 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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 08-14-21 and 08-24-21. 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 10-28-21 and 10-29-21 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, at least four compounds (Delta BHC, DDT, Methoxychlor and Endosulfan sulfate) failed the maximum 20% difference in the last daily standard analyzed on 10-28-21. Since the recoveries were all high biased and no pesticide was detected in any of the field samples, this should not affect the quality of data. 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 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. Raw data for the QC samples were matching the reported values.

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: Sample BGMW10102021 was identified as field duplicate of BGMW10102021D. Both sample and corresponding field duplicate sample were reported as non-detected for the entire pesticide compound list.

4.3.6. Raw data was submitted for all samples. Sample BGMW12102021 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. Three water samples were collected on 10-07-21, extracted on 10-13-21, and analyzed on 10-19-21.

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 10-14-21. 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 10-14-21. 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 would be injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-19-21, 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 channels.

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 method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD, therefore result 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: Sample BGMW10102021 was identified as field duplicate of BGMW10102021D. Both sample and corresponding field duplicate sample were reported as non-detected for Polychlorinated Biphenyls (PCBs).

4.4.6. Raw data was submitted for all samples. Sample BGMW12102021 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.

4.5. 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 10-07-21, extracted on 10-13-21 and analyzed on 11-05-21.

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 10-28-21. 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.

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 10-28-21. 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 11-05-21, 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 compounds from channels A. At least four compounds exceeded 20% limit in the closing daily standard in column B.

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 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 except for one sample (BGMW10102021), in which the surrogate recovery exceeded maximum permissible limit of 138% (recovery=192%). Since the recovery is high biased and no herbicide was detected in the sample, this should not affect data quality.

4.5.5. Field duplicate sample and its associated sample: Sample BGMW10102021 was identified as field duplicate of BGMW10102021D. Both sample and corresponding field duplicate sample were reported as non-detected for herbicides.

4.5.6. Raw data was submitted for all samples. Sample BGMW12102021 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.

4.6. 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 10-07-21, extracted on 10-12-21 and analyzed on 10-13-21 and 10-14-21, 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 08-24-21. 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 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% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of four continuing calibration standards were analyzed on 10-13-21 and 10-14-21, 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 method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. The 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**:** Sample BGMW10102021 was identified as field duplicate of sample BGMW10102021D. No explosive target compound was detected in sample and associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample BGMW12102021 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 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 10-07-21, extracted on 10-12-21 and analyzed on 10-13-21.

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 10-13-21, 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 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 within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Sample BGMW10102021 was identified as field duplicate of sample BGMW10102021D. No explosive target compound was detected in sample and field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample BGMW12102021 was selected to be reviewed as stage 3 data deliverable. Raw data of 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. 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 10-07-21. Samples were analyzed on 10-12-21 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 09-23-21. 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 09-23-21. 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 10-12-21, 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 collected for this method.

4.8.6. Raw data was submitted for all samples. Sample BGMW12102021 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 three water samples requested for this method. Water samples were collected on 10-07-21, extracted on 10-14-21 and analyzed on 10-15-21 within holding time.

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 08-12-21. 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 08-12-21. 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 10-15-21 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 collected for this method.

4.9.6. Raw data was submitted for all samples. Sample BGMW12102021 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 seven water samples requested for this method. Water samples were collected on 10-07-21. Samples were analyzed on 10-14-21 and 10-15-21 within 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 09-21-21. 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 0.999 (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 09-21-21. 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 10-14-21 and 10-15-21, 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 TMW15102021 was designated to be spiked as MS/MSD. Perchlorate 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 BGMW10102021 was identified as field duplicate of BGMW10102021D. No perchlorate was detected in either sample or associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample BGMW12102021 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 the data summary reports.

4.11. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.11.1. 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 10-07-21. Samples were prepared (digested) for both total and dissolved metals on 10-18-21. Samples and QC samples were analyzed on 11-12-21 and 11-15-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Two sets of initial calibration were established at the start of each analysis day on 11-12-21 and 11-15-21. Tune performance report was generated at the start of each analysis day, before initial calibration. It was within method's acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. The correlation coefficient of at least 0.999 was calculated to show the linearity of calibration curve for each metal. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

Metals	Concentration µg/L
Se, Pb, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Mn, Ni, Ag, Tl, V, and Zn	50,100,500,1000
Al, Fe, Ca, Mg, Na, and K	50,000

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of ±20% of the true value for the analytes incorporated in each solution.

4.11.3. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW15102021 was designated to be spiked as MS/MSD. Recoveries of LCS/LCSD were all within the acceptance limit of 80-120% for both total and dissolved metals. Recoveries of MS/MSD were mostly acceptable except two metals as listed in the table below. As a result, all the positive results for parent sample will be qualified as estimated value "J" for Sodium. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	TMW15102021 MS%	TMW15102021 MSD%	**QC Limit%**	TMW15102021 MS%	TMW15102021 MSD%	**QC Limit%**
Aluminum	√	√	84-117	√	√	84-117
Antimony	√	√	85-117	√	√	85-117
Arsenic	√	√	84-116	√	√	84-116
Barium	√	√	86-114	√	√	86-114
Beryllium	√	√	83-121	√	√	83-121
Cadmium	88	86*	87-115	√	√	87-115
Calcium	√	√	87-118	√	√	87-118
Chromium	√	√	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	√	√	85-118
Iron	√	√	87-118	√	√	87-118
Lead	√	√	88-115	√	√	88-115
Magnesium	√	√	83-118	√	√	83-118
Manganese	√	√	87-115	√	√	87-115
Nickel	√	√	85-117	√	√	85-117
Potassium	√	√	85-115	√	√	85-115
Selenium	√	√	80-120	√	√	80-120
Silver	√	√	85-116	√	√	85-116
Sodium	267*	467*	85-117	367*	-100*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

Method blank results for metals were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard. Sample TMW15102021 was used for serial dilution for both total and dissolved metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis. The same sample was used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total and dissolved metals.

4.11.4. Field duplicate sample and its associated sample: Sample BGMW10102021 was identified as field duplicate of BGMW10102021D. Results for sample/sample duplicate is summarized in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	BGMW10102021 µg/L	BGMW10102021D µg/L	% RPD	BGMW10102021 µg/L	BGMW10102021D µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	6.4	6.2	3.17	6.1	6.4	4.8
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	8100	8000	1.24	7900	8000	1.26
Chromium	8.3	U	200	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	U	U	--	U	U	--
Lead	U	U	--	U	U	--
Magnesium	930	910	2.17	900	910	1.10
Manganese	18	16	11.8	14	14	<1
Nickel	5.3	U	200	U	U	--
Potassium	720J	700J	2.82	740J	730J	1.36
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	640000	630000	1.57	630000	640000	1.57
Thallium	U	U	--	U	U	--
Vanadium	U	U	--	U	U	--
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Sample BGMW12102021 was selected to be reviewed as stage 3 data deliverable. Raw data of 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.

4.12. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.12.1. 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 10-07-21. Samples were prepared (digested) and analyzed for Mercury and dissolved Mercury on 10-18-21. All samples were preserved and filtered for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 10-18-21. The correlation coefficient of at least 0.999980 was calculated to show the linearity of calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

Initial calibration verification and Continuing calibration verification standards for mercury was within the acceptable range (90-110% of the spiked value).

4.12.3. Quality Control: The QC samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Sample TMW15102021 was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW15102021 was also used for serial dilution. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample BGMW10102021 was identified as field duplicate of BGMW10102021D. No Mercury was detected in sample and corresponding field duplicate sample for Mercury and dissolved Mercury analysis.

4.12.5. Raw data was submitted for all samples. Sample BGMW12102021 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 raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #21J090 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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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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SDG #21J092 Analytical Data Package

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 10-11-21. EMAX Laboratories received the samples on 10-12-21. The 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 MW23102021 (Lab ID#J092-06) 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 BGMW13S102021(Lab ID#J092-16) was designated to be spiked as MS/MSD. Raw data for this sample together with 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 (17 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 initial and re-analysis of all samples. The deviations, if any, are discussed in Section 4.0 for this method.

The SDG # 21J092 analytical data evaluated in this data validation report 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 seventeen (17) water samples were collected on 10-11-21. EMAX Laboratories received the samples on 10-12-21.

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 MW23102021 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 BGMW13S102021 was designated to be spiked as MS/MSD on the chain of custody. Raw data for this sample together with other QC samples in was also 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 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;
- LCSs 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#21J092 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
TMW53102021	J092-01	10-11-21	S3VM	Anions by IC,
MW24102021	J092-02	10-11-21	S3VM	Anions by IC,
MW24102021D	J092-03	10-11-21	S3VM	Anions by IC,
TMW52102021	J092-04	10-11-21	S3VM	Anions by IC,
MW23102021D	J092-05	10-11-21	S3VM	Anions by IC,
MW23102021	J092-06	10-11-21	S3VM	Anions by IC,
MW03102021	J092-07	10-11-21	S3VM	Anions by IC,
MW26102021	J092-08	10-11-21	S3VM	Anions by IC,
MW36D102021	J092-09	10-11-21	S3VM	Anions by IC,
TMW57102021	J092-10	10-11-21	S3VM	Anions by IC,
MW36S102021	J092-11	10-11-21	S3VM	Anions by IC,
TMW04102021	J092-12	10-11-21	S3VM	Anions by IC,
TMW59102021	J092-13	10-11-21	S3VM	Anions by IC,
MW33102021	J092-14	10-11-21	S3VM	Anions by IC,
MW34102021	J092-15	10-11-21	S3VM	Anions by IC,
BGMW13S102021	J092-16	10-11-21	S3VM	Anions by IC,
BGMW11102021	J092-17	10-11-21	S3VM	Anions by IC,
BGMW13S102021MS	J092-16M	10-11-21	S3VM	Anions by IC,
BGMW13S102021MSD	J092-16S	10-11-21	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 2.2˚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 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 9056A: Anions) =17/17X100=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 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, Chloride, Fluoride, Nitrate-N, Nitrite, Orthophosphate, and Sulfate

4.1.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of seventeen (17) water samples requested for this method. Water samples were collected on 10-11-21. Samples were analyzed on 10-12-21 and 10-13-21 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Samples for Bromide, Chloride, fluoride and Sulfate were analyzed and re-analyzed with dilution on 11-04-21, 11-05-21, within 28-day holding time. All initial analysis were within 28-day holding time for Bromide, Chloride, Fluoride and Sulfate.

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 on the basis of retention time as compared to reference standards.

Two instruments were initially calibrated with nine calibration levels (0.05-20mg/L) on 08-12-21 and 10-21-21. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout the analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. 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 08-12-21 and 10-21-21. Recoveries were all within 90-110% of expected true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-12-21, 10-13-21, 11-04-21, and 11-05-21. A total of seven continuing calibration standards were initially analyzed on 10-12-21 and 10-13-21. Later, nine more continuing calibration standards were analyzed with reference to separate initial calibrations on 11-04-21 and 11-05-21. 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.

4.1.3. Quality Control Samples consisted of one method blank and one set of LCS/LCSD, MS/MSD and sample/sample duplicate for initial analysis of all anions. Sample BGMW13S102021 was designated to be spiked as MS/MSD and sample/sample duplicate analysis. Two more sets of quality control samples (MB and LCS/LCD) were also analyzed with re-analysis and /or dilutions of samples for Bromide, Fluoride, Chloride, and Sulfate. Recoveries of LCS/LCSDs were all within 90-110 % of spiked values for each anion. Recoveries of MS/MSD were mostly within acceptable limits except for some anions as indicated in the table below:

Anion	BGMW13S102021 MS%	BGMW13S102021 MSD%	% Acceptance limits
Nitrite	84*	85*	87-111
Orthophosphate	79*	82	80-116
Nitrate	81*	82*	88-111
Bromide	61*	62*	91-110
Chloride	70*	48*	87-111
Sulfate	78*	77*	87-112

*Outside Acceptance limits

Therefore, the reported values will be qualified as estimated value (“J”) for positive results and as (“UJ”) for non-detect results in the parent sample for these anions. Percent RPDs were less than 20% for LCS/LCSD and MS/MSD. Sample BGMW13S102021 was also used for sample/sample duplicate analysis. All the recoveries and % RPDs were within acceptance limits for each anion.

4.1.4. Field duplicate sample and its associated sample: Sample MW24102021 was identified as field duplicate of MW24102021D and sample MW23102021 was identified as field duplicate of MW23102021D. Results for each set of sample/sample duplicate is summarized in the tables below:

Anion	MW24102021 (Lab ID #J092-02) mg/L	MW24102021D (Lab ID #J092-03) mg/L	**% RPD**
Nitrate	U	U	--
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.36J	0.36J	<1
Chloride	110	110	<1
Fluoride	0.97	0.98	1.02
Sulfate	2.5	1.6	43.9

Anion	MW23102021 (Lab ID #J092-06) mg/L	MW23102021D (Lab ID #J092-05) mg/L	**% RPD**
Nitrate	U	U	--
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.54	0.57	5.40
Chloride	97	86	12.1
Fluoride	0.78	0.76	2.60
Sulfate	3.9	4.2	7.41

4.1.5. Raw data was submitted for all requested field samples. Sample MW23102021 was designated to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met. Samples were analyzed and/or re-analyzed for Bromide, Fluoride, Sulfate and Chloride, on 11-04-21 and 11-05-21 within 28-day holding time.

5.0 CONCLUSION

SDG #21J092 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 #21J102 Analytical Data Package

Publication Date: 01-25-22

By
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TABLE OF CONTENTS

EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 10-12-21. EMAX Laboratories received the samples on 10-13-21. 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 TMW64102021(Lab ID# J102-04) was designated 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. Sample TMW64102021(Lab ID# J102-04) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with method blank and LCS/LCSD for each method 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, (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 (10 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (10 samples)
EPA Method SW8332: Nitroglycerine and PETN (10 samples)
EPA Method 8081B: Organochlorine Pesticides (8 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (3 sample)

EPA Method 8151A: Chlorinated herbicides (3 sample)

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)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (11 samples)

EPA Method 7470A: Mercury & Dissolved Mercury (11 samples)
EPA Method SW9056A: Anions (11 samples)

EPA Method 3520C/8270SIM: 1,4-Dioxane by GC/MS (1 sample)

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 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 # 21J102 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 twelve (12) water samples were collected on 10-12-21. EMAX Laboratories received the samples on 10-13-21.

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 TMW64102021 (EMAX ID #J102-04) from this sample delivery group was designated as stage 3 data deliverable. Raw data for this sample was evaluated comprehensively. Sample TMW64102021 (EMAX ID #J102-04) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with other QC samples in this report LCS/LCSD was also 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#21J102 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW14A102021	21J102-01	10-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Anions by IC
QC12102021TB7	21J102-02	10-12-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW14A102021D	21J102-03	10-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Anions by IC
TMW64102021	21J102-04	10-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW45102021	21J102-05	10-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21J102 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW45102021D	J102-06	10-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
MW25102021	J102-07	10-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW28102021	J102-08	10-12-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Anions by IC
FW31102021	J102-09	10-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Anions by IC
TMW31D102021	J102-10	10-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21J102 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW43102021	J102-11	10-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
QC12102021EB4	J102-12	10-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC 1,4-Dioxane by 8270SIM
TMW64102021MS	J102-04M	10-12-21	S3VM	Nitroaromatics and Nitramines Nitroglycerine & PETN Semivolatiles + APP9
TMW64102021MSD	J102-04S	10-12-21	S3VM	Nitroaromatics and Nitramines Nitroglycerine & PETN Semivolatiles + APP9

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
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	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	0.5µg/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	None. Holding times were met
Mercury and Dissolved Mercury	Water	Collection to Analysis: 28 days	None. Holding times were met
Anions by IC	Water	Analysis 48 hours from collection For Nitrate, Nitrite and Orthophosphate; 28 days from collection for the rest	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 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 3.0˚C and as high as 5.8˚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:

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

Completeness (EPA Method 5030B/8260C: VOCs) =12/12X100=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) =8/8X100=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) 4/4X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =3/3X100=100%
Completeness (EPA Method 6850: Perchlorate) =7/7X100=100%
Completeness (EPA Method 7470A: Mercury and Dissolved Mercury) =11/11X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =11/11X100=100%
Completeness (EPA Method SW9056A: Anions) =11/11X100=100%
Completeness (EPA Method 3520B/8270SIM: 1,4-Dioxane) =1/1X100=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
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury and Dissolved Mercury by Cold Vapor
EPA **Method 9056A** for Anions by IC
EPA Method **3520C/8270** SIM for 1,4-Dioxane by GC/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 10-12-21. Samples were analyzed on 10-13-21 and 10-14-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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 4.1.7.

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 10-06-21 and at the beginning of analysis shift on 10-13-21. 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 10-06-21. 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 10-06-21
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.

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

Target Analytes	Least Square Linear Regression (CCF) 10-06-21
Chloroform	0.9991

Average response factors for all target compounds were within method's recommended values, except for: Acetone (0.025) 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 10-06-21
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 10-06-21. 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 compound except for Chloroform (21.0%).

Continuing calibration check standard was analyzed at the beginning and end of analysis shift on 10-13-21 and 10-14-21. Prior to continuing calibration analysis, **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 10-13-21 (I)	Continuing cal. Response factors 10-14-21 (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 (10-13-21) I	%Deviation from Initial calibration (10-14-21) 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 the initial calibration was less than 20 percent for the rest of target list, except for two compounds listed in the table below. This should not affect the data quality.

Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-13-21) Opening	%Deviation from Initial calibration (10-14-21) Closing
Vinyl acetate	≤ 20%	28.1*	25.3*
2-Chloroethyl vinyl ether	≤ 20%	25.7*	30.7*

4.1.5. Quality Control samples reported consisted of one method blank and one set of LCS/LCSD. No sample was designated to be analyzed 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: Sample TMW14A102021 was identified as field duplicate of sample TMW14A102021D, and sample TMW45102021was identified as field duplicate of sample TMW45102021D. Both samples and corresponding field duplicate samples were reported as non-detected for all volatile organic compound list.

4.1.7. Raw data was submitted for all samples. Sample TMW64102021 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. Traces of Bromodichloromethane, Bromoform and Dibromochloromethane was detected in equipment blank. However, these compounds were not detected in any of the field samples.

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

4.2.1. Technical Holding Times

Holding time requirement was met for ten samples. Water samples were collected on 10-12-21, extracted on 10-18-21 and were analyzed on 10-20-21 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 08-12-21, 08-13-21 and at the beginning of analysis shift on 10-20-21. 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 08-12-21 and 08-13-21. 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 08-12-21
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 15%) 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 08-12-21
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) 08-12-21
Benzoic acid	0.9991
2,4-Dinitrophenol	0.9993
4-Nitrophenol	1.00
4,6-Dinitro-2-methylphenol	0.9983
Benzidine	0.9994
3,3-Dimethyl benzidine	0.9999

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

4.2.4. Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard on 08-12-21 and 08-13-21. 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 except 4,6-Dinitro-2-methylphenol (25.0%). Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-20-21 and 10-21-21. 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 (10-20-21) I	Continuing cal. Response factors (10-21-21) 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 nearly 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 (10-20-21) I	Accepted Deviation from Initial calibration (10-21-21) 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.

4.2.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD Sample TMW64102021 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 within the project acceptance limits for the entire compound list in LCS/LCSD. Recoveries of MS/MSD were mostly within QC acceptable limits except for compounds listed in the table below.

Target compound	TMW64102021MS	TMW64102021MSD	**QC Limit%**
2-Nitrophenol	70%	46*%	47-123
Phenol	70%	47*%	50-130

Therefore, the results of above compounds will be qualified as “UJ” in the parent sample.

Quite a number of compounds failed the maximum %RPD in MS/MSD.

The results and recoveries of LCS/LCSD was used to evaluate accuracy and precision for 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: Sample TMW14A102021 was identified as field duplicate of TMW14A102021D and sampleTMW45102021 was identified as field duplicate of TMW45102021D. Both samples and corresponding field duplicate samples were reported as non-detected for the entire Semi-Volatile Compound list.

4.2.7. Raw data was submitted for all samples. Sample TMW64102021 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 10-12-21, extracted on 10-15-21 and analyzed on 10-26-21and 10-27-21 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 08-23-21 and before sample analysis on 10-26-21 and 10-27-21. 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 08-23-21. 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 08-14-21 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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 08-14-21 and 08-24-21. 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 10-26-21 and 10-27-21 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, at least three compounds (DDD, DDT, and Endosulfan sulfate) failed the maximum 20% difference in daily standards analyzed on 10-27-21 in column A. Since the recoveries were all high biased and no pesticide was detected in any of the field samples, this should not affect the quality of data. 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 one 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. Raw data for the QC samples were matching the reported values.

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: Sample TMW45102021 was identified as field duplicate of TMW45102021D. Both sample and corresponding field duplicate sample were reported as non-detected for the entire pesticide compound list.

4.3.6. Raw data was submitted for all samples. Sample TMW64102021 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.

4.4. 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 three water samples requested for this method. Water samples were collected on 10-12-21, extracted on 10-15-21, and analyzed on 10-19-21.

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 10-14-21. 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 10-14-21. 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 would be injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-19-21 and 10-20-21, 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 both channels.

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

4.4.6. Raw data was submitted for all samples. Sample TMW64102021 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.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for three water samples requested for this method. Water samples were collected on 10-12-21, extracted on 10-15-21 and analyzed on 10-29-21.

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 10-28-21. 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.

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 10-28-21. 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 10-29-21, 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 compounds from channels A.

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. The 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 TMW64102021 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.

4.6. 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 10-12-21, extracted on 10-19-21 and analyzed on 10-21-21, 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 a different column (Kinetex- Biphenyl column).

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-24-21. 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 08-24-21. 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 six continuing calibration standards (both primary and confirmation columns) were analyzed on 10-20-21, 10-21-21and 10-22-21, 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 and MS/MSD. Sample TMW14A102021 was selected to be spiked as MS/MSD. The full explosive target list was 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 TMW14A102021 was identified as field duplicate of sample TMW14A102021D and sample TMW45102021 was identified as field duplicate of sample TMW45102021D. No explosive target compound was detected in each sample and corresponding field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample TMW64102021 was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data of 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. All positive results, if any were confirmed with confirmation 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 10-12-21, extracted on 10-19-21 and analyzed on 10-21-21.

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 10-21-21 and 10-22-21, 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 TMW14A102021 was selected 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 within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Sample TMW14A102021 was identified as field duplicate of sample TMW14A102021D and sample TMW45102021 was identified as field duplicate of sample TMW45102021D. No explosive target compound was detected in each sample and corresponding field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample TMW64102021 was designated to be reviewed as stage 3 data deliverable. Raw data of 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. 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 10-12-21. Samples were analyzed on 10-14-21 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 09-09-21. 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 09-09-21. 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 10-14-21 and 10-15-21, 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 collected for this method.

4.8.6. Raw data was submitted for all samples. Sample TMW64102021 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.

4.9. 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 samples requested for this method. Water samples were collected on 10-12-21, extracted on 10-19-21 and analyzed on 10-20-21 within holding time.

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 08-12-21. 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 08-12-21. 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 10-20-21 and 10-21-21 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 collected for this method.

4.9.6. Raw data was submitted for all samples. Sample TMW64102021 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 samples requested for this method. Seven (7) water samples were collected on 10-12-21. Samples were analyzed on 10-14-21 and 10-15-21 within 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 09-21-21. 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 0.999 (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 09-21-21. 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 10-14-21 and 10-15-21, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were all 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. Perchlorate was spiked and reported for each set of 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 TMW45102021 was identified as field duplicate of TMW45102021D. No perchlorate was detected in either sample or associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample TMW64102021 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.

4.11. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.11.1. 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 10-12-21. Samples were prepared (digested) for both total and dissolved metals on 10-18-21. Samples and QC samples were analyzed on 11-12-21 and 11-15-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Two sets of initial calibration were established at the start of each analysis day on 11-12-21 and 11-15-21. Tune performance report was generated at the start of each analysis day, before initial calibration. It was within method's acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. The correlation coefficient of at least 0.999 was calculated to show the linearity of calibration curve for each metal. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

Metals	**Concentration µg/L**
Se, Pb, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Mn, Ni, Ag, Tl, V, and Zn	50,100,500,1000
Al, Fe, Ca, Mg, Na, and K	50,000

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of ±20% of the true value for the analytes incorporated in each solution.

4.11.3. Quality Control samples consisted of one method blank and one set of LCS/LCSD for total and dissolved metals. No sample was spiked as MS/MSD. Recoveries of LCS/LCSD were all within the acceptance limit of 80-120% for both total and dissolved metals.

Method blank results for metals were acceptable and no contamination was found in the method blanks except traces of lead (0.18J, at <½LOQ). Calibration blanks were analyzed after each continuing calibration standard.

4.11.4. Field duplicate sample and its associated sample: Sample TMW14A102021 was identified as field duplicate of TMW14A102021D and sample TMW45102021 was identified as field duplicate of TMW45102021D. Results for each set of sample/sample duplicate is summarized in the tables below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW14A102021 µg/L	TMW14A102021D µg/L	% RPD	TMW14A102021 µg/L	TMW14A102021D µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	20	19	5.13	15	16	6.45
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	3900	3700	5.26	3300	3300	<1
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	U	U	--	U	U	--
Lead	U	U	--	U	U	--
Magnesium	430J	420J	2.35	390J	390J	<1
Manganese	18	16	11.8	9.1	9.0	1.10
Nickel	u	U	--	U	U	--
Potassium	530J	510J	3.85	510J	500J	1.98
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	450000	440000	2.55	440000	440000	<1
Thallium	U	U	--	U	U	--
Vanadium	U	U	--	U	U	--
Zinc	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	TMW45102021 µg/L	TMW45102021D µg/L	% RPD	TMW45102021 µg/L	TMW45102021D µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	TMW45102021 µg/L	TMW45102021D µg/L	% RPD	TMW45102021 µg/L	TMW45102021D µg/L	% RPD
Arsenic	U	U	--	U	U	--
Barium	59	58	1.71	56	55	1.8
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	29000	28000	3.51	28000	28000	<1
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	U	U	--	U	U	--
Lead	U	U	--	U	U	--
Magnesium	7800	7600	2.60	7500	7400	1.34
Manganese	130	110	16.7	53	51	3.85
Nickel	U	U	--	U	U	--
Potassium	460J	480J	4.26	530J	440J	18.6
Selenium	1.0J	0.87J	13.9	0.80J	0.95J	17.1
Silver	U	U	--	U	U	--
Sodium	950000	960000	1.05	930000	910000	2.17
Thallium	U	U	--	U	U	--
Vanadium	4.3J	4.8J	11	4.5J	4.2J	6.90
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Sample TMW64102021 was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data of 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.

4.12. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.12.1. 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 10-12-21. Samples were prepared (digested) and analyzed for Mercury and dissolved Mercury on 10-26-21. All samples were preserved and filtered for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 10-26-21. The correlation coefficient of at least 0.999753 was calculated to show the linearity of calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

Initial calibration verification and Continuing calibration verification standards for mercury was within the acceptable range (90-110% of the spiked value).

4.12.3. Quality Control: The QC samples consisted of one method blank, LCS/LCSD and MS/MSD for Mercury. Sample TMW28102021 was selected to be spiked as MS/MSD. No MS/MSD sample was analyzed for dissolved Mercury. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW28102021 was also used for serial dilution for Mercury. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in each method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample TMW14A102021 was identified as field duplicate of TMW14A102021D and sample TMW45102021 was identified as field duplicate of TMW45102021D. No Mercury was detected in each sample and corresponding field duplicate sample for Mercury and dissolved Mercury.

4.12.5. Raw data was submitted for all samples. Sample TMW64102021 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 raw data, agreed with all the results reported in data summary reports.

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

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of water samples requested for this method. Eleven (11) water samples were collected on 10-12-21. Samples were analyzed on 10-13-21 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Samples for Chloride, Sulfate and fluoride were re-analyzed with dilution on 11-04-21 and 11-05-21 within 28-day holding time. Samples for Bromide were analyzed on 11-05-21 within 28-day holding time.

4.13.2. 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 on the basis of retention time as compared to reference standards.

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 08-12-21. 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 component. 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 initial calibration on 08-12-21. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-13-21, 11-04-21 and 11-05-21. A total of nine continuing calibration standards were analyzed with re-analysis of some 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.

4.13.3. Quality Control Samples consisted of one method blank and one set of LCS/LCSD for Nitrate, Nitrite and Orthophosphate. No sample was designated to be spiked as MS/MSD. A second set of QCs (MB and LCS/LCD) was analyzed with dilutions and/or re-analysis of samples for Bromide, Chloride, Fluoride and Sulfate. Recoveries of LCS/LCSD were all within 90-110 % of spiked values for each anion. Percent RPDs were less than 20% for LCS/LCSD.

4.13.4. Field duplicate sample and its associated sample: Sample TMW14A102021 was identified as field duplicate of TMW14A102021D and sample TMW45102021 was identified as field duplicate of TMW45102021D. Results foreach set of sample/sample duplicate is summarized in the tables below:

ANALYTE	Anions		
	TMW14A102021 mg/L	TMW14A102021D mg/L	% RPD
Nitrate	U	U	--
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.67	0.67	<1
Chloride	160	170	6.60
Fluoride	0.46	0.46	<1
Sulfate	350	350	<1

ANALYTE	Anions		
	TMW45102021 mg/L	TMW45102021D mg/L	% RPD
Nitrate	1.2	1.3	8.0
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.45J	0.45J	<1
Chloride	200	220	9.52
Fluoride	0.60	0.60	<1
Sulfate	550	590	7.02

4.13.5. Raw data was submitted for all requested field samples. Sample TMW64102021 was designated to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met. Due to high concentrations of Sulfate and Chloride, samples were analyzed with high dilution factors on 11-04-21 and 11-05-21 within 28-day holding time.

4.14. 1,4-Dioxane (EPA Method 3520C/8270SIM)

4.14.1. Technical Holding Times

Holding time requirement was met for one water sample. Water sample was collected on 10-12-21, extracted on 10-15-21 and were analyzed on 10-18-21; within holding time.

Selective Ion Monitoring (SIM) method with GC/MS was used for analysis.

Isotopically-labeled 1,4-Dioxane-d8 was added to serve as internal standard.

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 4.14.7.

4.14.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 08-04-21 and at the beginning of analysis batch on 10-18-21. It passed all the method assigned criteria.

4.14.3. Initial Calibration

Sample and QC samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 08-04-21. A multi-level calibration standard ranging from 0.05mg/L to 50mg/L was used for this purpose. Internal standard curve type in Selective Ion Monitoring (SIM) mode was used for initial calibration and all following analysis. 1,4-Dioxane-d8 was used as internal standard. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table. Average response curve type was used for 1,4-Dioxane and Bromobenzene as surrogate. Target compounds met the maximum 15% RSD limit (all less than 15%).

4.14.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 08-04-21. Percent difference (%D) were within ±20% of the expected value.

Continuing calibration check standard was analyzed at the beginning and at the end of analysis shift on 10-18-21. Prior to opening continuing calibration standard, instrument performance tune check standard (BFB) was carried out. It passed all the method tuning criteria. Percent difference (%D) and/or drift for both opening and closing continuing calibration standards were within ±20% of the expected value for each daily standard.

4.14.5. Quality Control: The QC samples reported consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. 1,4-Dioxane as the only target with Bromobenzene as surrogate was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits.

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 method’s acceptance limit.

4.14.6. **Field duplicate sample** and its associated sample: No field duplicate sample was assigned for this method.

4.14.7. Sample QC12102021EB4 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 #21J102 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 #21J103 Analytical Data Package

Publication Date: 01-26-22

By
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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 nineteen (19) water samples were collected on 10-10-21 and 10-11-21. EMAX Laboratories received the samples on 10-13-21. 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 MW23102021(Lab ID# J103-08) was designated as stage 3 data deliverable. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation. Sample TMW27102021(Lab ID# J103-19) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with method blank and LCS/LCSD for each method 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, (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 (19 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (16 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (17 samples)
EPA Method SW8332: Nitroglycerine and PETN (17 samples)
EPA Method 8081B: Organochlorine Pesticides (15 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (11 samples)
EPA Method 8151A: Chlorinated herbicides (11 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (13 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (12 samples)
EPA Method 6850: Perchlorate (18 samples)
EPA Method SW6020A: Dissolved and total Metals by ICP-MS (18 samples)
EPA Method 7470A: Mercury & Dissolved Mercury (18 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 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 # 21J103 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 nineteen (19) water samples were collected on 10-10-21 and 10-11-21. EMAX Laboratories received the samples on 10-13-21.

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 MW23102021 (EMAX ID #J103-08) from this sample delivery group was designated as stage 3 data deliverable. Raw data for this sample was evaluated comprehensively. Sample TMW27102021 (EMAX ID #J103-19) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with other QC samples in this report LCS/LCSD was also 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#21J103 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW52102021	21J103-01	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
MW36D102021	21J103-02	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
MW36S102021	21J103-03	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
MW26102021	21J103-04	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls

Site Name: Fort Wingate, New Mexico

SDG#21J103 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
MW24102021	J103-05	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
TMW53102021	J103-06	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
QC11102021TB6	J103-07	10-11-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
MW23102021	J103-08	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850
MW23102021D	J103-09	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#21J103 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW24102021D	J103-10	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
TMW57102021	J103-11	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
MW33102021	J103-12	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
MW03102021	J103-13	10-11-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline; TPH as DRO Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#21J103 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW59102021	J103-14	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
MW34102021	J103-15	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
TMW04102021	J103-16	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850
BGMW13S102021	J103-17	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls

Site Name: Fort Wingate, New Mexico

SDG#21J103 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
BGMW11102021	J103-18	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
TMW27102021	J103-19	10-10-21	S3VM	VOCs by SW5030B/8260C, Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850
TMW27102021MS	J103-19M	10-10-21	S3VM	VOCs by SW5030B/8260C, Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850
TMW27102021MSD	J103-19S	10-10-21	S3VM	VOCs by SW5030B/8260C, Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS 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
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	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	0.5µg/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-eight 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	None. Holding times were met
Mercury and Dissolved Mercury	Water	Collection to Analysis: 28 days	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 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-eight ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler’s temperature was reported as low as 2.1˚C and as high as 4.9˚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:

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

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) =19/19X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =16/16X100=100%
Completeness (EPA Method 8330B: Explosives) =17/17X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =17/17X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =15/15X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =11/11X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =11/11X100=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) =18/18X100=100%
Completeness (EPA Method 7470A: Mercury and Dissolved Mercury) =18/18X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =18/18X100=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 nineteen 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 **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury and Dissolved Mercury by Cold Vapor

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. Nineteen water samples were collected on 10-10-221 and 10-11-21. Samples were analyzed on 10-14-21, 10-18-21, 10-19-21 and 10-20-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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 4.1.7.

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 initial calibration on 10-06-21, 10-11-21 and at the beginning of each analysis shift on 10-14-21, 10-18-21, 10-19-21 and 10-20-21. 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 10-06-21 and 10-11-21. 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 10-06-21	Average Response factor 10-11-21
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.

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) 10-06-21	Least Square Linear Regression (CCF) 10-11-21
Chloroform	0.9991	NA
Vinyl acetate	NA	0.9982

Average response factors for all target compounds were within method’s recommended values, except for: Acetone (0.025) and 2-Butanone (0.015) in initial calibration analyzed on 10-06-21. 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 10-06-21	Response Factors %RSD 10-11-21
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 10-06-21 and 10-12-21. 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 compound except for Chloroform (21.0%, ICV run on 10-06-21), and Vinyl acetate (30.2% ICV run on 10-12-21). Continuing calibration check standards were analyzed at the beginning and end of each analysis shift on 10-15-21, 10-18-21, 10-19-21 and 10-20-21. Prior to continuing calibration analysis, **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 10-14-21 (I&II)	Continuing cal. Response factors 10-18-21 (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

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-19-21 (I&II)	Continuing cal. Response factors 10-20-21 (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 (10-14-21) I&II	%Deviation from Initial calibration (10-18-21) 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

Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-19-21) I&II	%Deviation from Initial calibration (10-20-21) 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 the initial calibration was less than 20 percent for the rest of target list, except for the compounds listed in the tables below. This should not affect the data quality.

Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-14-21)		%Deviation from Initial calibration (10-18-21)	
		I	II	I	II
2-Chloroethylvinyl ether	≤ 20%	31.4*	36.4*	26.4*	37.1*
Tetrehydrofuran	≤ 20%	--	38.9*	--	33.3*
Benzene	≤ 20%		33.9*	--	--

*Outside acceptance limits

Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-19-21)		%Deviation from Initial calibration (10-20-21)	
		I	II	I	II
2-Chloroethylvinyl ether	≤ 20%	36.4*	37.1*	25.9*	26.5*
Tetrehydrofuran	≤ 20%	31.5*	35.2*	--	--
Chloroethane	≤ 20%	--	20.6*	--	--
Dichlorofluoromethane	≤ 20%	--	24.4*	--	24.3*
Trichlorofluoromethane	≤ 20%	--	27.0*	--	--
Acetone	≤ 20%	--	28.0*	--	--

* Outside acceptance limits

4.1.5. Quality Control samples reported consisted of four method blanks, four sets of LCS/LCSD and MS/MSD. Sample TMW27102021was designated to be analyzed 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: Sample MW24102021 was identified as field duplicate of sample MW24102021D, and sample MW23102021was identified as field duplicate of sample MW23102021D. Both sample (MW24102021) and corresponding field duplicate sample were reported as non-detected for all volatile organic compounds list.

Traces of Acetone was detected in sample MW23102021 and corresponding field duplicate sample as shown in the table below:

Target compound	MW23102021 µg/L	MW23102021D µg/L	%RPD
Acetone	5.3	5.6	5.50

4.1.7. Raw data was submitted for all samples. Sample MW23102021 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 sixteen samples. Water samples were collected on 10-11-21, extracted on 10-16-21 and were analyzed on 10-19-21 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 08-17-21, 08-18-21 and at the beginning of analysis shift on 10-19-21. 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 08-17-21 and 08-18-21. 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 08-17-21
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 15%) 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 08-17-21
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) 08-17-21**
Benzoic acid	0.9963
2,4-Dinitrophenol	0.9988

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

4.2.4. Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard on 08-17-21 and 08-18-21. 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 10-19-21. 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 (10-19-21) 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 nearly 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 (10-19-21) 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 compound listed in the table below in the closing daily calibration standard:

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (10-19-21) closing
Caprolactam	≤ 20	28.6*

*Exceeded 20% maximum allowed difference in the closing daily standard

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 for the entire compound list in LCS/LCSD. The results and recoveries of LCS/LCSD was used to evaluate accuracy and precision for 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: Sample MW24102021 was identified as field duplicate of MW24102021D and sample MW23102021 was identified as field duplicate of MW23102021D. Both samples and corresponding field duplicate samples were reported as non-detected for the entire Semi-Volatile Compound list.

4.2.7. Raw data was submitted for all samples. Sample MW23102021 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 fifteen water samples requested for this method. Water samples were collected on 10-11-21, extracted on 10-16-21 and analyzed on 11-01-21and 11-02-21 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 08-23-21 and before sample analysis on 11-01-21 and 11-02-21. 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 08-23-21. 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 08-14-21 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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 08-14-21 and 08-24-21. 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 11-01-21 and 11-02-21 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, at least four compounds (Delta BHC, DDD, DDT, and Endosulfan sulfate) failed the maximum 20% difference in the closing daily standard analyzed on 11-01-21 in column A. Since the recoveries were all high biased and no pesticide was detected in any of the field samples, this should not affect the quality of data. 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 one 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. Raw data for the QC samples were matching the reported values.

Result for method blank was 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: Sample MW24102021 was identified as field duplicate of MW24102021D and sample MW23102021 was identified as field duplicate of sample MW23102021D. Both samples and corresponding field duplicate samples were reported as non-detected for the full pesticide compound list.

4.3.6. Raw data was submitted for all samples. Sample MW23102021 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 eleven water samples requested for this method. Water samples were collected on 10-11-21, extracted on 10-16-21, and analyzed on 10-19-21 and 10-20-21.

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 10-14-21. 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 10-14-21. 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 would be injected at single point for identification only.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-19-21 and 10-20-21, 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 both channels.

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 method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD, therefore result 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 assigned to this method.

4.4.6. Raw data was submitted for all samples. Sample TMW52102021 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.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eleven water samples requested for this method. Water samples were collected on 10-11-21, extracted on 10-18-21 and analyzed on 11-01-21 and 11-02-21.

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 10-28-21. 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.

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 10-28-21. 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.

Five continuing calibration standards were analyzed at 10-injections interval. It was carried out on 11-01-21 and 11-02-21, 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 compounds from channels A except Dicamba (21%; column A) in the closing daily standard. This minor exceedance should not affect data quality.

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 list of herbicides target compounds 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 TMW52102021 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.

4.6. 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 seventeen (17) water samples requested for this method. Water samples were collected on 10-11-21, extracted on 10-18-21 and analyzed on 10-19-21 and 10-20-21, 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 08-23-21 and 08-24-21. 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 08-24-21. 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 six continuing calibration standards (both primary and confirmation columns) were analyzed on 10-19-21 and 10-20-21, bracketing the analyses of samples and all the QC samples. Two daily standards were analyzed with Biphenyl column (confirmation column) on 10-22-21. 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. The full 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 MW24102021 was identified as field duplicate of sample MW24102021D and sample MW23102021 was identified as field duplicate of sample MW23102021D. No explosive target compound was detected in each sample and corresponding field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample MW23102021 was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data of 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. All positive results, if any were confirmed with confirmation 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 seventeen (17) water samples requested for this method. Water samples were collected on 10-11-21, extracted on 10-18-21 and analyzed on 10-19-21.

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. Confirmation 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. 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 08-23-21 and 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 10-19-21, bracketing the analyses of samples and all the QC samples. To confirm positive results, two daily standards were also analyzed with confirmation column on 10-22-21. 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 within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Sample MW24102021 was identified as field duplicate of sample MW24102021D and sample MW23102021 was identified as field duplicate of sample MW23102021D. No explosive target compound was detected in each sample and corresponding field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample MW23102021 was designated to be reviewed as stage 3 data deliverable. Raw data of 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. 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 10-11-21. Samples were analyzed on 10-14-21 and 10-15-21 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 09-09-21. 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 09-09-21. 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 10-14-21 and 10-15-21, 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 MW36S102021 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 sample was collected for this method.

4.8.6. Raw data was submitted for all samples. Sample TMW52102021 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 twelve water samples requested for this method. Water samples were collected on 10-11-21, extracted on 10-15-21 and analyzed on 10-18-21 and 10-19-21 within holding time.

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 08-12-21. 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 08-12-21. 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 10-18-21 and 10-19-21 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 assigned to this method.

4.9.6. Raw data was submitted for all samples. Sample TMW52102021 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 all sample requested for this method. Eighteen (18) water samples were collected on 10-10-21 and 10-11-21. Samples were analyzed on 10-15-21 within 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 09-21-21. 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 0.999 (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 09-21-21. 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 10-15-21, 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 TMW27102021 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 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 MW24102021 was identified as field duplicate of MW24102021D and sample MW23102021 was identified as field duplicate of MW23102021D. No perchlorate was detected in either samples or associated field duplicate samples.

4.10.6. Raw data was submitted for all samples. Sample MW23102021 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.

4.11. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eighteen water samples requested for this method. Water samples were collected on 10-10-21 and 10-11-21. Samples were prepared (digested) for both total and dissolved metals on 10-18-21. Samples and QC samples were analyzed on 11-10-21 and 11-11-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Two sets of initial calibration were established at the start of each analysis day on 11-10-21 and 11-11-21. Tune performance report was generated at the start of each analysis day, before initial calibration. It was within method’s acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. The correlation coefficient of at least 0.999 was calculated to show the linearity of calibration curve for each metal. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

Metals	Concentration µg/L
Se, Pb, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Mn, Ni, Ag, Tl, V, and Zn	50,100,500,1000
Al, Fe, Ca, Mg, Na, and K	50,000

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of ±20% of the true value for the analytes incorporated in each solution.

4.11.3. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW27102021 was spiked as MS/MSD. Recoveries of LCS/LCSD were all within the acceptance limit of 80-120% for both total and dissolved metals. Recoveries of MS/MSD were mostly acceptable except few metals listed in the table below. As a result, all the positive results for parent sample will be qualified as estimated value “J” for these metals. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	TMW27102021 MS%	TMW27102021 MSD%	QC Limit%	TMW27102021 MS%	TMW27102021 MSD%	QC Limit%
Aluminum	√	√	84-117	√	√	84-117
Antimony	√	√	85-117	√	√	85-117
Arsenic	√	√	84-116	√	√	84-116
Barium	√	√	86-114	√	√	86-114
Beryllium	√	√	83-121	√	√	83-121
Cadmium	√	√	87-115	86*	84*	87-115
Calcium	130*	117	87-118	120*	87	87-118
Chromium	√	√	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	√	√	85-118
Iron	√	√	87-118	√	√	87-118
Lead	√	√	88-115	√	√	88-115
Magnesium	√	√	83-118	√	√	83-118
Manganese	153*	117*	87-115	117*	103	87-115
Nickel	√	√	85-117	√	√	85-117

ANALYTE	Total Metals			Dissolved Metals		
	TMW27102021 MS%	TMW27102021 MSD%	**QC Limit%**	TMW27102021 MS%	TMW27102021 MSD%	**QC Limit%**
Potassium	√	√	85-115	√	√	85-115
Selenium	√	√	80-120	√	√	80-120
Silver	√	√	85-116	√	√	85-116
Sodium	400*	267*	85-117	67*	133*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

Method blank results for metals were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard. Sample TMW27102021 was used for serial dilution for both total and dissolved metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis. The same sample was used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total and dissolved metals.

4.11.4. Field duplicate sample and its associated sample: Sample MW24102021 was identified as field duplicate of MW24102021D and sample MW23102021 was identified as field duplicate of MW23102021D. Results for each set of sample/sample duplicate is summarized in the tables below:

ANALYTE	Total Metals			Dissolved Metals		
	MW24102021 (Lab ID# J103-05) µg/L	MW24102021D (Lab ID# J103-10) µg/L	**% RPD**	MW24102021 (Lab ID# J103-05) µg/L	MW24102021D (Lab ID# J103-10) µg/L	**% RPD**
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	0.71J	0.85J	17.9	0.76J	0.77J	1.31
Barium	310	290	6.67	290	290	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	32000	32000	<1	32000	32000	<1
Chromium	0.13J	0.13J	<1	U	0.10J	200
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	1800	1800	<1	1800	1800	<1
Lead	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	MW24102021 (Lab ID# J103-05) µg/L	MW24102021D (Lab ID# J103-10) µg/L	**% RPD**	MW24102021 (Lab ID# J103-05) µg/L	MW24102021D (Lab ID# J103-10) µg/L	**% RPD**
Magnesium	11000	11000	<1	11000	11000	<1
Manganese	470	430	8.89	440	430	2.30
Nickel	U	U	--	U	U	--
Potassium	730	750	2.70	740	740	<1
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	260000	260000	<1	260000	260000	<1
Thallium	U	U	--	U	U	--
Vanadium	0.47J	0.85J	57.6	0.58J	0.68J	15.9
Zinc	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	MW23102021 (Lab ID# J103-08) µg/L	MW23102021D (Lab ID# J103-09) µg/L	**% RPD**	MW23102021 (Lab ID# J103-08) µg/L	MW23102021D (Lab ID# J103-09) µg/L	**% RPD**
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	1.6J	1.6J	<1	1.4J	1.4J	<1
Barium	220	230	4.44	210	210	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	17000	17000	<1	17000	16000	6.06
Chromium	U	11	200	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	160J	210J	27.0	U	U	--
Lead	U	0.42J	200	U	U	--
Magnesium	7000	7100	1.42	7100	7100	<1
Manganese	73	76	4.03	67	67	<1
Nickel	U	15	200	U	U	--
Potassium	1000	1000	<1	1000	1000	<1
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	460000	460000	<1	460000	470000	2.15
Thallium	U	U	--	U	U	--
Vanadium	4.8J	5.2J	8.0	4.1J	4.2J	2.41
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Sample MW23102021 was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data of 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.

4.12. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.12.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eighteen (18) water samples requested for this method. Water samples were collected on 10-10-21 and 10-11-21. Samples were prepared (digested) and analyzed for Mercury and dissolved Mercury on 10-26-21. All samples were preserved and filtered for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 10-26-21. The correlation coefficient of at least 0.999753 was calculated to show the linearity of calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

Initial calibration verification and continuing calibration verification standards for mercury was within the acceptable range (90-110% of the spiked value).

4.12.3. Quality Control: The QC samples consisted of one method blank one set of LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Sample TMW27102021 was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW27102021 was also used for serial dilution for Mercury and dissolved Mercury. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in each method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample MW24102021 was identified as field duplicate of MW24102021D and sample MW23102021 was identified as field duplicate of MW23102021D. No Mercury was detected in each of the samples and corresponding field duplicate samples for Mercury and dissolved Mercury.

4.12.5. Raw data was submitted for all samples. Sample MW23102021 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 raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #21J103 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 #21J117 Analytical Data Package

Publication Date: 01-26-22

By
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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 10-13-21. EMAX Laboratories received the samples on 10-14-21. 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 was submitted for the requested analytical methods. Sample MW31102021(Lab ID# J117-06) was designated 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. No sample was designated to be spiked as MS/MSD on the chain of custody. Therefore, the results and recoveries of LCS/LCSD were used to evaluate both accuracy and precision. Raw data for method blank and LCS/LCSD for each method 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, (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 (9 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (12 samples)
EPA Method SW8332: Nitroglycerine and PETN (12 samples)
EPA Method 8081B: Organochlorine Pesticides (9 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (6 sample)
EPA Method 8151A: Chlorinated herbicides (6 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (11 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (10 samples)

EPA Method 6850: Perchlorate (15 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (16 samples)

EPA Method 7470A: Mercury & Dissolved Mercury (16 samples)
EPA Method SW9056A: Anions by IC (16 samples)

EPA Method 3520C/8270SIM: 1,4-Dioxane by GC/MS (2 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 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 # 21J117 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 seventeen (17) water samples were collected on 10-13-21. EMAX Laboratories received the samples on 10-14-21.

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 MW31102021 (EMAX ID #J117-06) from this sample delivery group was designated 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 QC samples, method blanks and LCS/LCSD was also 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 presented
- 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#21J117 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW61102021	21J117-01	10-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
QC13102021TB8	21J117-02	10-13-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW34102021	21J117-03	10-13-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline; TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW25102021	21J117-04	10-13-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Anions by IC
TMW34102021D	21J117-05	10-13-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline; TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21J117 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
MW31102021	J117-06	10-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
MW37102021	J117-07	10-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC 1,4-Dioxane by 8270SIM
TMW17102021	J117-08	10-13-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
MW18102021	J117-09	10-13-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline; TPH as DRO Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21J117 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW38102021	J117-10	10-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
MW35102021	J117-11	10-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW13102021	J117-12	10-13-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
MW35102021D	J117-13	10-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21J117 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW39D102021	J117-14	10-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
TMW21102021	J117-15	10-13-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Anions by IC
QC13102021EB5	J117-16	10-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC 1,4-Dioxane by 8270SIM
MW36102021	J117-17	10-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	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	0.5µg/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-two 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	None. Holding times were met
Mercury and Dissolved Mercury	Water	Collection to Analysis: 28 days	None. Holding times were met
Anions by IC	Water	Analysis 48 hours from collection For Nitrate, Nitrite and Orthophosphate; 28 days from collection for the rest	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 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-two ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler’s temperature was reported as low as 4.1˚C and as high as 5.9˚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:

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

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) =17/17X100=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) =9/9X100=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) 11/11X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =10/10X100=100%
Completeness (EPA Method 6850: Perchlorate) =15/15X100=100%
Completeness (EPA Method 7470A: Mercury and Dissolved Mercury) =16/16X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =16/16X100=100%
Completeness (EPA Method SW9056A: Anions by IC) =16/16X100=100%
Completeness (EPA Method 3520B/8270SIM: 1,4-Dioxane) =2/2X100=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
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury and Dissolved Mercury by Cold Vapor
EPA **Method 9056A** for Anions by IC
EPA Method **3520C/8270** SIM for 1,4-Dioxane by GC/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 seventeen samples. Water samples were collected on 10-13-21. Samples were analyzed on 10-19-21 and 10-22-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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 4.1.7.

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 initial calibration on 10-06-21, 10-11-21 and at the beginning of each analysis shift on 10-19-21and 10-22-21. 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 10-06-21 and 10-11-21. 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 10-06-21	Average Response factor 10-11-21
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.

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) 10-06-21	Least Square Linear Regression (CCF) 10-11-21
Chloroform	0.9991	NA
Vinyl acetate	NA	0.9982

Average response factors for all target compounds were within method's recommended values, except for: Acetone (0.025) and 2-Butanone (0.015) in initial calibration analyzed on 10-06-21. However, all 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 10-06-21	Response Factors %RSD 10-11-21
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 set of initial calibration was verified by a second source standard on 10-06-21 and 10-12-21. 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 compound except for Chloroform (21.0% in ICV run on 10-06-21), and Vinyl acetate (30.2% in ICV run on 10-12-21).

Continuing calibration check standards were analyzed at the beginning and end of each analysis shift on 10-19-21 and 10-22-21. Prior to continuing calibration analysis, **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 10-19-21 (I&II)	Continuing cal. Response factors 10-22-21 (I&II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

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 (10-19-21) I&II	%Deviation from Initial calibration (10-22-21) 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 the initial calibration was less than 20 percent for the rest of target list, except for the compounds listed in the tables below. This should not affect the data quality.

Compounds	**%Deviation From Initial calibration (Acceptance Limit)**	**%Deviation from Initial calibration (10-19-21)**		**%Deviation from Initial calibration (10-22-21)**	
		I	II	I	II
2-Chloroethylvinyl ether	≤ 20%	36.4*	37.1*	32.9*	24.1*
Chloroethane	≤ 20%	--	20.6*	--	--
Dichlorofluoromethane	≤ 20%	--	24.4*	--	--
Trichlorofluoromethane	≤ 20%	--	27.0*	--	--
Acetone	≤ 20%	--	28.0*	--	--
Vinyl acetate	≤ 20%	--	--	25.3*	26*

* Outside QC limits

4.1.5. Quality Control samples reported consisted of two method blanks and two sets of LCS/LCSD. No sample was designated to be analyzed 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: Sample TMW34102021 was identified as field duplicate of sample TMW34102021D, and sample MW35102021was identified as field duplicate of sample MW35102021D. Both sample (MW35102021) and corresponding field duplicate sample were reported as non-detected for all volatile organic compounds list.

Traces of Acetone was detected in sample TMW34102021D as shown in the table below:

Target compound	TMW34102021 µg/L	TMW34102021D µg/L	**%RPD**
Acetone	U	6.0J	200

4.1.7. Raw data was submitted for all samples. Sample MW31102021 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. The results calculated from the raw data agreed with all the results reported in data summary reports.

Traces of Bromodichloromethane, Bromoform, Chloroform and Dibromochloromethane was detected in equipment blank. However, these compounds were not detected in any of the field samples.

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 10-13-21, extracted on 10-20-21 and were analyzed on 10-22-21 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 08-12-21, 08-13-21 and at the beginning of analysis shift on 10-22-21. 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 08-12-21 and 08-13-21. 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 08-12-21
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 15%) 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 08-12-21
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. All analytes met the acceptance criteria regarding minimum response factor and maximum %RSD.

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) 08-12-21
Benzoic acid	0.9991
2,4-Dinitrophenol	0.9993
4-Nitrophenol	1.00
4,6-Dinitro-2-methylphenol	0.9983
Benzidine	0.9994
3,3-Dimethyl benzidine	0.9999

4.2.4. Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard on 08-12-21 and 08-13-21. 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 except 4,6-Dinitro-2-methylphenol (25.0%). Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-22-21. 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 (10-22-21) 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 nearly 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 (10-22-21) 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 listed below in the closing continuing calibration standard:

Analyte	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (10-22-21) I	%Deviation from Initial calibration (10-22-21) II
4,6-Dinitro-2-methylphenol	≤ 20	22.0*	22.0*
2-Nitroaniline	≤ 20	--	22.2*

*Exceeded 20% maximum allowed difference in the closing daily standard

This minor exceedance should not affect 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 all the QC samples reported were within the project acceptance limits for the entire compound list in LCS/LCSD, except for 1,2,4,5-Tetrachlorobenzene (RPD 24%) and Benzaldehyde (RPD 33%). The results and recoveries of LCS/LCSD was used to evaluate both accuracy and precision for 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: Sample MW35102021 was identified as field duplicate of MW35102021D. Both sample and corresponding field duplicate sample were reported as non-detected for the full Semi-Volatile Compound list.

4.2.7. Raw data was submitted for all samples. Sample MW31102021 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 nine water samples requested for this method. Water samples were collected on 10-13-21, extracted on 10-20-21 and analyzed on 11-04-21 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 08-23-21 and before sample analysis on 11-04-21. 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 08-23-21. 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 08-14-21 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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 08-14-21 and 08-24-21. 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 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 11-04-21 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, at least three compounds (DDD, DDT, and Endosulfan sulfate) failed the maximum 20% difference in the closing daily standard in column A. Since the recoveries were all high biased and no pesticide was detected in any of the field samples, this should not affect the quality of data. 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 one 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. Raw data for the QC samples were matching the reported values.

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: Sample MW35102021 was identified as field duplicate of MW35102021D. Both sample and corresponding field duplicate sample were reported as non-detected for the entire pesticide compound list.

4.3.6. Raw data was submitted for all samples. Sample MW31102021 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 six water samples requested for this method. Water samples were collected on 10-13-21, extracted on 10-20-21, and analyzed on 10-25-21.

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 10-14-21. 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 10-14-21. 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 would be injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-25-21, 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 both channels.

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 result 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: Sample MW35102021 was identified as field duplicate of MW35102021D. Both sample and corresponding field duplicate sample were reported as non-detected for Polychlorinated Biphenyls (PCBs).

4.4.6. Raw data was submitted for all samples. Sample MW31102021 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.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six water samples requested for this method. Water samples were collected on 10-13-21, extracted on 10-19-21 and analyzed on 11-01-21 and 11-02-21.

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 10-28-21. 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.

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 10-28-21. 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 11-01-21 and 11-02-21, 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 compounds from channels A. Recovery of Dicamba was high biased in two mid daily standards for channel B.

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: Sample MW35102021 was identified as field duplicate of MW35102021D. Both sample and corresponding field duplicate sample were reported as non-detected for all chlorinated herbicides.

4.5.6. Raw data was submitted for all samples. Sample MW31102021 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.

4.6. 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

10-13-21, extracted on 10-20-21 and analyzed on 10-21-21 and 10-22-21, 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 08-23-21 and 08-24-21. 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 08-24-21. 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 five continuing calibration standards (both primary and confirmation columns) were analyzed on 10-21-21 and 10-22-21, 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 selected 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 MW35102021 was identified as field duplicate of sample MW35102021D. No explosive target compound was detected in each sample and corresponding field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample MW31102021 was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data of 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. All positive results were confirmed with Biphenyl confirmation 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 10-13-21, extracted on 10-20-21 and analyzed on 10-21-21.

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 10-21-21, 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 within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Sample MW35102021 was identified as field duplicate of sample MW35102021D. No explosive target compound was detected in each sample and corresponding field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample MW31102021 was designated to be reviewed as stage 3 data deliverable. Raw data of 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. 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 eleven water samples requested for this method. Water samples were collected on 10-13-21. Samples were analyzed on 10-15-21 and 10-16-21 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 09-09-21. 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 09-09-21. 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 10-15-21 and 10-16-21, 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: Sample TMW34102021 was identified as field duplicate of sample TMW34102021 and sample MW35102021 was identified as field duplicate of sample MW35102021D. No TPH as Gasoline was detected in each sample and corresponding field duplicate sample.

4.8.6. Raw data was submitted for all samples. Sample MW31102021 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 ten water samples requested for this method. Water samples were collected on 10-13-21, extracted on 10-20-21 and analyzed on 10-22-21 within holding time.

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 08-12-21. 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 08-12-21. 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 10-21-21 and 10-22-21 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: Sample TMW34102021 was identified as field duplicate of sample TMW34102021 and sample MW35102021 was identified as field duplicate of sample MW35102021D. No TPH as Diesel was detected in each sample and corresponding field duplicate sample.

4.9.6. Raw data was submitted for all samples. Sample MW31102021 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 samples requested for this method. Fifteen (15) water samples were collected on 10-13-21. Samples were analyzed on 10-19-21 and 10-20-21 within 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 09-21-21. 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 0.999 (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 09-21-21. 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 analyzed on 10-19-21 and 10-20-21, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were all within 85-115% limit.

4.10.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW36102021was selected to be spiked as MS/MSD. perchlorate was spiked and reported for LCS/LCSD and 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 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 TMW34102021 was identified as field duplicate of sample TMW34102021D and sample MW35102021 was identified as field duplicate of sample MW35102021D.

Results for sample/sample duplicates are summarized in the tables below:

Analyte	TMW34102021 (Lab ID #J117-03) µg/L	TMW34102021D (Lab ID #J117-05) µg/L	**%RPD**
Perchlorate	0.19J	0.16J	17.1

Analyte	MW35102021 (Lab ID #J117-11) µg/L	MW35102021D (Lab ID #J117-13) µg/L	**%RPD**
Perchlorate	U	U	--

4.10.6. Raw data was submitted for all samples. Sample MW31102021 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.

4.11. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for sixteen water samples requested for this method. Water samples were collected on 10-13-21. Samples were prepared (digested) for both total and dissolved metals on 10-19-21. Samples and QC samples were analyzed on 11-19-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

One set of initial calibration was established at the start of analysis day on 11-19-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method's acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. The correlation coefficient of at least 0.999 was calculated to show the linearity of calibration curve for each metal. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

Metals	**Concentration µg/L**
Se, Pb, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Mn, Ni, Ag, Tl, V, and Zn	50,100,500,1000
Al, Fe, Ca, Mg, Na, and K	50,000

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of ±20% of the true value for the analytes incorporated in each solution.

4.11.3. Quality Control samples consisted of one method blank and one set of LCS/LCSD for total and dissolved metals. No sample was designated to be spiked as MS/MSD. Recoveries of LCS/LCSD were all within the acceptance limit of 80-120% for both total and dissolved metals.

Method blank results for metals were acceptable and no contamination was found in the method blanks except traces of Lead (<1/2 LOQ) in method blank for total metals. Calibration blanks were analyzed after each continuing calibration standard. All results were within the QC limit of (80-120%) for total and dissolved metals.

4.11.4. Field duplicate sample and its associated sample: Sample TMW34102021 was identified as field duplicate of TMW34102021D and sample MW35102021 was identified as field duplicate of MW35102021D. Results for each set of sample/sample duplicate is summarized in the tables below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW34102021 (Lab ID #J117-03) µg/L	TMW34102021D (Lab ID #J117-05) µg/L	**% RPD**	TMW34102021 (Lab ID #J117-03) µg/L	TMW34102021D (Lab ID #J117-05) µg/L	**% RPD**
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	11	11	--	9.9J	11	10.5
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	130000	130000	<1	120000	120000	<1
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	U	U	--	U	U	--
Lead	U	U	--	U	U	--
Magnesium	27000	27000	<1	26000	27000	3.77
Manganese	200	190	5.13	120	120	<1
Nickel	U	U	--	U	U	--
Potassium	620J	580J	6.66	700J	630J	10.5
Selenium	120	120	<1	120	120	<1
Silver	U	U	--	U	U	--
Sodium	1400000	1300000	7.41	1300000	1300000	<1
Thallium	U	U	--	U	U	--
Vanadium	U	U	--	U	U	--
Zinc	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	MW35102021 (Lab ID #J117-11) µg/L	MW35102021D (Lab ID #J117-13) µg/L	**% RPD**	MW35102021 (Lab ID #J117-11) µg/L	MW35102021D (Lab ID #J117-13) µg/L	**% RPD**
Aluminum	210J	210J	<1	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	13	12	8.00	9.3	9.3	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	20000	20000	<1	19000	20000	5.13
Chromium	0.86J	U	200	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	130J	U	200	U	U	--
Lead	U	U	--	U	U	--
Magnesium	2800	2800	<1	2700	2700	<1
Manganese	43	44	2.30	37	37	<1
Nickel	U	U	--	U	U	--
Potassium	1100	1100	<1	1100	1200	8.70
Selenium	0.95J	U	200	U	U	--
Silver	U	U	--	U	U	--
Sodium	770000	760000	1.31	750000	760000	1.32
Thallium	U	U	--	U	U	--
Vanadium	3.0J	1.6J	60.9	U	1.6J	200
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Sample MW31102021 was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data of 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.

4.12. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.12.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for sixteen (16) water samples requested for this method. Water samples were collected on 10-13-21. Samples were prepared (digested) and analyzed for Mercury and dissolved Mercury on 10-27-21. All samples were preserved and filtered for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 10-27-21. The correlation coefficient of at least 0.999911 was calculated to show the linearity of calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

Initial calibration verification and Continuing calibration verification standards for mercury was within the acceptable range (90-110% of the spiked value).

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of method blank, LCS/LCSD and MS/MSD for Mercury. Sample TMW13102021 was selected to be spiked as MS/MSD for Mercury and dissolved Mercury. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW13102021 was also used for serial dilution. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in each method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample TMW34102021 was identified as field duplicate of TMW34102021D and sample MW35102021 was identified as field duplicate of MW35102021D. No Mercury was detected in each sample and corresponding field duplicate sample for Mercury and dissolved Mercury.

4.12.5. Raw data was submitted for all samples. Sample MW31102021 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 raw data, agreed with all the results reported in data summary reports.

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

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of sixteen water samples requested for this method. Water samples were collected on

10-13-21. Samples were analyzed on 10-14-21 and 10-15-21 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Samples for Bromide, Chloride, Fluoride and Sulfate were initially analyzed on 10-14-21, 10-15-21 and re-analyzed with dilution on 11-08-21 and 11-09-21 within 28-day holding time. All initial analysis and/or re-analysis were within required holding time.

4.13.2. 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 on the basis of retention time as compared to reference standards.

Two instruments were initially calibrated with nine calibration levels (0.05-20mg/L) on 08-12-21 and 10-21-21. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout the analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. 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 08-12-21 and 10-21-21. Recoveries were all within 90-110% of expected true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-14-21,10-15-21, 11-08-21 and 11-09-21. A total of ten continuing calibration standards were analyzed with re-analysis of some samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values, except for Orthophosphate in one daily standard as indicated in the table below:

Compounds	**(Acceptance Limit) %**	**(10-14-21) III**
Orthophosphate	90-110	89.6*

*Outside control limit

After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions.

4.13.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for Nitrate, Nitrite, Orthophosphate, Bromide, Fluoride, Chloride and Sulfate. Sample TMW36102021 was selected to be spiked as MS/MSD and sample/sample duplicate analysis. A second set of QCs (MB and LCS/LCD) was analyzed with re-analysis and/or dilutions of samples for Bromide, Chloride, Fluoride and Sulfate. Recoveries of LCS/LCSDs were all within 90-110 % of spiked values for each anion. Percent RPDs were less than 20% for LCS/LCSD. However, recoveries of MS/MSD failed the acceptance limits for few anions as detailed in the table below:

Anion	TMW36102021 MS%	TMW36102021 MSD%	Acceptance limits%
Nitrate	79*	80*	88-111
Orthophosphate	56*	57*	80-116
Bromide	37*	37*	91-110
Chloride	89	85*	87-111

Therefore, the positive results will be qualified as estimated value (“J”) and non-detected anions will be qualified as “UJ” in the parent sample.

Sample TMW36102021 was also used for sample/sample duplicate analysis. Percent RPDs were all within QC acceptance criteria (less than 20%).

4.13.4. Field duplicate sample and its associated sample: Sample TMW34102021 was identified as field duplicate of TMW34102021D and sample MW35102021 was identified as field duplicate of MW35102021D. Results for each set of sample/sample duplicate is summarized in the tables below:

Anion	TMW34102021 (Lab ID #J117-03) mg/L	TMW34102021D (Lab ID #J117-05) mg/L	**% RPD**
Nitrate	66	66	--
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.78	0.78	<1
Chloride	260	250	3.92
Fluoride	U	U	--
Sulfate	1500	1600	6.45

Anion	MW35102021 (Lab ID #J117-11) mg/L	MW35102021D (Lab ID #J117-13) mg/L	% RPD
Nitrate	U	U	--
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.56	0.56	<1
Chloride	330	260	23.7
Fluoride	0.16	0.15	6.45
Sulfate	990	910	8.42

4.13.5. Raw data was submitted for all requested field samples. Sample MW31102021 was designated to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met. Due to high concentrations of Sulfate and Chloride, samples were analyzed with high dilution factors on 11-08-21and 11-09-21 within 28-day holding time.

4.14. 1,4-Dioxane (EPA Method 3520C/8270SIM)

4.14.1. Technical Holding Times

Holding time requirement was met for two water samples. Water samples were collected on 10-13-21, extracted on 10-15-21 and were analyzed on 10-18-21; within holding time.

Selective Ion Monitoring (SIM) method with GC/MS was used for analysis.

Isotopically-labeled 1,4-Dioxane-d8 was added to serve as internal standard.

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 4.14.7.

4.14.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 08-04-21 and at the beginning of analysis batch on 10-18-21. It passed all the method assigned criteria.

4.14.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 08-04-21. A multi-level calibration standard ranging from 0.05mg/L to 50mg/L was used for this purpose. Internal standard curve type in Selective Ion Monitoring (SIM) mode was used for initial calibration and all following analysis. 1,4-Dioxane-d8 was used as internal standard. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table. Average response curve type was used for 1,4-Dioxane and Bromobenzene as surrogate. Target compounds met the maximum 15% RSD limit (all less than 15%).

4.14.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 08-04-21. Percent difference (%D) were within ±20% of the expected value.

Continuing calibration check standard was analyzed at the beginning and at the end of analysis shift on 10-18-21. Prior to opening continuing calibration standard, instrument performance tune check standard (BFB) was carried out. It passed all the method tuning criteria. Percent difference (%D) and/or drift for both opening and closing continuing calibration standards were within ±20% of the expected value for each daily standard.

4.14.5. Quality Control: The QC samples reported consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. 1,4-Dioxane as the only target with Bromobenzene as surrogate was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits.

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 method’s acceptance limit.

4.14.6. **Field duplicate sample** and its associated sample: No field duplicate sample was assigned for this method.

4.14.7. Sample MW37102021 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 #21J117 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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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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SDG #21J126 Analytical Data Package

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 ten (10) water samples were collected on 10-14-21. EMAX Laboratories received the samples on 10-15-21. The 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 MW22D102021 (Lab ID#J126-10) 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. Sample BGMW13D102021 (Lab ID#J126-08) was designated to be spiked as MS/MSD. Raw data for this sample together with 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 (10 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 analytical holding times were met for initial and re-analysis of all samples. The deviations, if any, are discussed in Section 4.0 for this method.

The SDG # 21J126 analytical data evaluated in this data validation report 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 ten (10) water samples were collected on 10-14-21. EMAX Laboratories received the samples on 10-15-21.

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 MW22D102021 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. Sample BGMW13D102021 was designated to be spiked as MS/MSD on the chain of custody. Raw data for this sample together with other QC samples in was also 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 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;
- LCSs 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#21J126 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
MW38102021	J126-01	10-14-21	S3VM	Anions by IC,
MW39102021	J126-02	10-14-21	S3VM	Anions by IC,
TMW06102021	J126-03	10-14-21	S3VM	Anions by IC,
TMW06102021D	J126-04	10-14-21	S3VM	Anions by IC,
TMW03102021	J126-05	10-14-21	S3VM	Anions by IC,
TMW24102021	J126-06	10-14-21	S3VM	Anions by IC,
TMW32102021	J126-07	10-14-21	S3VM	Anions by IC,
BGMW13D102021	J126-08	10-14-21	S3VM	Anions by IC,
TMW47102021	J126-09	10-14-21	S3VM	Anions by IC,
MW22D102021	J126-10	10-14-21	S3VM	Anions by IC,
BGMW13D102021MS	J126-08M	10-14-21	S3VM	Anions by IC,
BGMW13D102021MSD	J126-08S	10-14-21	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 4.2˚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 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:

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

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 9056A: Anions) =10/10X100=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 ten 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, Chloride, Fluoride, Nitrate-N, Nitrite, Orthophosphate and Sulfate

4.1.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of ten (10) water samples requested for this method. Water samples were collected on 10-14-21. Samples were analyzed on 10-15-21 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Some samples for Bromide, Chloride, fluoride and Sulfate were analyzed and/or re-analyzed with dilution on 11-09-21, within 28-day holding time.

4.1.2. Initial and continuing calibration: Anions such as Bromide, Chloride, Fluoride, 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 on the basis of retention time as compared to reference standards.

One instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 08-12-21. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout the 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 08-12-21. Recoveries were all within 90-110% of expected true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-15-21 and 11-09-21. A total of five continuing calibration standards were initially analyzed on 10-15-21. Later, three more continuing calibration standards were analyzed 11-09-21. 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 before each initial calibration. It was within the assigned QC limit for each anion.

4.1.3. Quality Control Samples consisted of one method blank and one set of LCS/LCSD, MS/MSD and sample/sample duplicate for initial analysis of all anions. Sample BGMW13D102021was designated to be spiked as MS/MSD and sample/sample duplicate analysis. A second set of MB and LCS/LCD was also analyzed with re-analysis and /or dilutions of samples for Bromide, Fluoride, Chloride, and Sulfate. Recoveries of LCS/LCSDs were all within 90-110 % of spiked values for each anion. Recoveries of MS/MSD were within acceptable limits except for some anions as indicated in the table below:

Anion	BGMW13D102021 MS%	BGMW13D102021 MSD%	% Acceptance limits
Orthophosphate	80	77*	80-116
Nitrate	81*	79*	88-111
Bromide	45*	45*	91-110
Chloride	84*	93	87-111
Sulfate	79*	89	87-112

*Outside Acceptance limits

Therefore, the reported values for these anions will be qualified as estimated value (“J”) for positive results and as (“UJ”) for non-detect results in the parent sample. Percent RPDs were less than 20% for LCS/LCSD and MS/MSD. Sample BGMW13D102021 was also used for sample/sample duplicate analysis. All the recoveries and % RPDs were within acceptance limits for each anion.

4.1.4. Field duplicate sample and its associated sample: Sample TMW06102021 was identified as field duplicate of TMW06102021D. Results for sample/sample duplicate is summarized in the table below:

Anion	TMW06102021 (Lab ID #J126-03) mg/L	TMW06102021D (Lab ID #J126-04) mg/L	**% RPD**
Nitrate	10	10	<1
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.42J	0.42J	<1
Chloride	120	59	68.2
Fluoride	U	U	--
Sulfate	610	610	<1

4.1.5. Raw data was submitted for all requested field samples. Sample MW22D102021 was selected to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met. Samples were analyzed and/or re-analyzed for Bromide, Fluoride, Sulfate and Chloride, on 11-09-21 within 28-day holding time requirement.

5.0 CONCLUSION

SDG #21J126 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 #21J145 Analytical Data Package

Publication Date: 01-26-22

By
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TABLE OF CONTENTS

EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 four (4) water samples were collected on 10-15-21. EMAX Laboratories received the samples on 10-16-21. 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 TMW48102021(Lab ID# J145-01) 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. 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 to evaluate 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 (4 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (3 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (3 samples)
EPA Method SW8332: Nitroglycerine and PETN (3 samples)
EPA Method 8081B: Organochlorine Pesticides (3 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (1 samples)
EPA Method 6850: Perchlorate (3 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (3 samples)

EPA Method 7470A: Mercury & Dissolved Mercury (3 samples)
EPA Method SW9056A: Anions by IC (3 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 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 # 21J145 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 four (4) water samples were collected on 10-15-21. EMAX Laboratories received the samples on 10-16-21.

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 present
- 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 TMW48102021 (EMAX ID #J145-01) from this sample delivery group was selected 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 blanks and LCS/LCSD was reviewed for each method. 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 present
- 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#21J145 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW48102021	21J145-01	10-15-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW40D102021	21J145-02	10-15-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850 Anions by IC
QC15102021TB10	21J145-03	10-15-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW49102021	21J145-04	10-15-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850 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	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	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	0.5µg/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
Organochlorine Pesticides	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
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	None. Holding times were met
Mercury and Dissolved Mercury	Water	Collection to Analysis: 28 days	None. Holding times were met
Anions by IC	Water	Analysis 48 hours from collection For Nitrate, Nitrite and Orthophosphate; 28 days from collection for the rest	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 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 1.3˚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) =4/4X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =3/33X100=100%
Completeness (EPA Method 8330B: Explosives) =3/3X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =3/3X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =3/3X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 1/1X100=100%
Completeness (EPA Method 6850: Perchlorate) =3/3X100=100%
Completeness (EPA Method 7470A: Mercury and Dissolved Mercury) =3/3X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =3/3X100=100%
Completeness (EPA Method SW9056A: Anions by IC) =3/3X100=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 four 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 **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 **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury and Dissolved Mercury by Cold Vapor
EPA **Method 9056A** for Anions 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. Four water samples were collected on 10-15-21. Samples were analyzed on 10-18-21within method's requirement for holding time. (Water samples were preserved with hydrochloric acid).

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 4.1.7.

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 10-12-21 and at the beginning of analysis shift on 10-18-21. 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 10-12-21. 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 10-12-21
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.

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) 10-12-21
Vinyl acetate	0.9961
2-Chloroethyl vinyl ether	0.9986
1,2-Dibromo-3-chloropropane	0.9995

Average response factors for all target compounds were within method's recommended values, except for: Acetone (0.050) 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 10-12-21
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 10-14-21. 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 compound except for Vinyl acetate (25.6%).

Continuing calibration check standards were analyzed at the beginning and end of analysis shift on 10-18-21. Prior to continuing calibration analysis, **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 10-18-21 (I)	Continuing cal. Response factors 10-18-21 (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 (10-18-21) I	%Deviation from Initial calibration (10-18-21) 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 the initial calibration was less than 20 percent for the rest of target list, except for the compound shown in the table below.

Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-18-21)	
		I	II
Chloromethane	≤ 20%	√	23.2*

*Outside QC limits

4.1.5. Quality Control samples reported consisted of one method blank and one set of LCS/LCSD. No sample was designated to be analyzed 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: No field duplicate sample was assigned to this sample delivery group.

4.1.7. Raw data was submitted for all samples. Sample TMW48102021 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 samples. Three water samples were collected on 10-15-21, extracted on 10-20-21 and were analyzed on 10-22-21and 10-23-21 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 08-12-21, 08-13-21 and at the beginning of analysis shift on 10-22-21. 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 08-12-21 and 08-13-21. 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 08-12-21
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 08-12-21
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) 08-12-21
Benzoic acid	0.9991
2,4-Dinitrophenol	0.9993
4-Nitrophenol	1.00
4,6-Dinitro-2-methylphenol	0.9983
Benzidine	0.9994
3,3, -Dimethylbenzidine	0.9999

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

4.2.4. Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard on 08-12-21 and 08-13-21. 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 except 4,6-Dinitro-2-methylphenol (25.0%). Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-22-21. 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 (10-22-21) I	Continuing cal. Response factors (10-23-21) 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 nearly 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 (10-22-21) I	Accepted Deviation from Initial calibration (10-23-21) 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	√	√

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (10-22-21) I	Accepted Deviation from Initial calibration (10-23-21) II
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 listed below in the opening and closing continuing calibration standards:

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (10-22-21) I	%Deviation from Initial calibration (10-23-21) II
4,6-Dinitro-2-methylphenol	≤ 20	22.0*	22.0*
Nitroaniline	≤ 20	--	22.2*

*Exceeded 20% maximum allowed difference in the closing daily standard

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 for the entire compound list in LCS/LCSD. The results and recoveries of LCS/LCSD was used to evaluate accuracy and precision for 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 sample was collected with this sample delivery group.

4.2.7. Raw data was submitted for all samples. Sample TMW48102021 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 three water samples requested for this method. Water samples were collected on 10-15-21, extracted on 10-22-21 and analyzed on 11-04-21and 11-05-21 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 08-23-21 and before sample analysis on 11-04-21 and 11-05-21. 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 08-23-21. 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 08-14-21 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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 full pesticide target list including Toxaphene, on 08-14-21 and 08-24-21. 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 11-04-21 and 11-05-21 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, at least three compounds (DDD, DDT, and Endosulfan sulfate) failed the maximum 20% difference in the last two closing daily standards analyzed on 11-05-21 in column A. Since the recoveries were all high biased and no pesticide was detected in any of the field samples, this should not affect the quality of data. 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 one 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. Raw data for the QC samples were matching the reported values.

Result for method blank was 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 all samples. Sample TMW48102021 was selected 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. 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 three (3) water samples requested for this method. Water samples were collected on 10-15-21, extracted on 10-21-21 and analyzed on 10-25-21, 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 08-23-21 and 08-24-21. 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 08-24-21. 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 (both primary and confirmation columns) were analyzed on

10-25-21 and 10-26-21, bracketing the analyses of samples and all the QC samples. Two daily standards were analyzed with Biphenyl column (confirmation column) on 10-26-21. 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 and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. The 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.

4.4.5 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 TMW48102021 was selected to be reviewed as stage 3 data deliverable on the chain of custody. Raw data of 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. All positive results, if any were confirmed with confirmation 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 three (3) water samples requested for this method. Water samples were collected on 10-15-21, extracted on 10-21-21 and analyzed on 10-21-21.

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. Confirmation 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. 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 08-23-21 and 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 10-21-21, 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 and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Each target analyte 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 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 TMW48102021 was designated to be reviewed as stage 3 data deliverable. Raw data of 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.

4.6. 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 water sample requested for this method. One (1) water sample was collected on 10-15-21. Sample was analyzed on 10-19-21 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 09-09-21. 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 09-09-21. 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 10-19-21, 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 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 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: No field duplicate sample was collected for this sample delivery group.

4.6.6. Sample QC15102021TB10 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.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for three samples requested for this method. Water samples were collected on 10-15-21. Samples were analyzed on 10-20-21 within holding time.

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

4.7.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 09-21-21. 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 0.999 (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.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 09-21-21. 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 analyzed on 10-19-21 and 10-20-21, bracketing the analyses and re-analysis of samples and all the QC samples. Recoveries of continuing calibration standards were all within 85-115% limit.

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. Perchlorate was spiked and reported for LCS/LCSD. 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 for each compound and no target was found in the method blank. No surrogate is used in this method.

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 TMW48102021 was selected 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.

4.8. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

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 10-15-21. Samples were prepared (digested) for both total and dissolved metals on 10-22-21. Samples and QC samples were analyzed on 11-17-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.8.2. Initial Calibration and Continuing calibration standards

One set of initial calibration was established at the start of analysis day on 11-17-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method's acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. The correlation coefficient of at least 0.999 was calculated to show the linearity of calibration curve for each metal. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

Metals	Concentration µg/L
Se, Pb, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Mn, Ni, Ag, Tl, V, and Zn	50,100,500,1000
Al, Fe, Ca, Mg, Na, and K	50,000

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of ±20% of the true value for the analytes incorporated in each solution.

4.8.3. Quality Control samples consisted of one method blank and one set of LCS/LCSD for total and dissolved metals. No sample was designated to be spiked as MS/MSD. Recoveries of LCS/LCSD were all within the acceptance limit of 80-120% for both total and dissolved metals.

Method blank results for metals were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard.

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

4.8.5. Raw data was submitted for all samples. Sample TMW48102021 was selected to be reviewed as stage 3 data deliverable on the chain of custody. Raw data of 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. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for three (3) water samples requested for this method. Water samples were collected on 10-15-21. Samples were prepared (digested) and analyzed for Mercury and dissolved Mercury on 10-29-21. Samples were analyzed on 10-30-21 within holding time. All samples were preserved and filtered for dissolved Mercury analysis.

4.9.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 10-30-21. The correlation coefficient of at least 0.999561 was calculated to show the linearity of calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L. Initial calibration verification and Continuing calibration verification standards for mercury was within the acceptable range (90-110% of the spiked value).

4.9.3. Quality Control: The QC samples consisted of one method blank and one set of LCS/LCSD for Mercury and dissolved Mercury. No sample was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD. Recoveries of LCS/LCSD and were within acceptable range of 82-119%. Method blank data was reviewed and no Mercury contamination was found in each method blanks.

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

4.9.5. Raw data was submitted for all samples. Sample TMW48102021 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 raw data, agreed with all the results reported in data summary reports.

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

4.10.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of three water samples requested for this method. Water samples were collected on 10-15-21. Samples were analyzed on 10-16-21 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Samples for Bromide, Chloride, Fluoride and Sulfate were analyzed/re-analyzed with dilution on 11-08-21 and 11-09-21 within 28-day holding time.

4.10.2. Initial and continuing calibration: Anions such as Bromide, Chloride, Fluoride, 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 on the basis of retention time as compared to reference standards.

Two instruments were initially calibrated with nine calibration levels (0.05-20mg/L) on 08-12-21 and 10-21-21. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout the analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each anon. 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 08-12-21 and 10-21-21. Recoveries were all within 90-110% of expected true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-16-21, 11-08-21 and 11-09-21. A total of eight continuing calibration standards were analyzed with re-analysis of some samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values, except Orthophosphate, as shown in the table below:

Compounds	**(Acceptance Limit) %**	**(10-16-21) I**	**(10-16-21) II**	**(10-16-21) III**
Orthophosphate	90-110	74.4*	73.3*	77.8*

*Outside control limit

As a result, all non-detect results will be qualified as estimated value “UJ”.

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 in each 24-hour. It was within the assigned QC limit for each anion.

4.10.3. Quality Control Samples consisted of one method blank and one set of LCS/LCSD for Nitrate, Nitrite, Orthophosphate, Bromide, Fluoride, Chloride and Sulfate. No sample was designated to be spiked as MS/MSD and sample/sample duplicate analysis. A second set of QCs (MB and LCS/LCD) was analyzed with dilutions/re-analysis of samples for Bromide, Chloride, Fluoride and Sulfate. Recoveries of LCS/LCSDs were all within 90-110 % of spiked values for each anion, except for Orthophosphate as indicated in the table below.

Anion	LCS%	LCSD%	% Acceptance limits
Orthophosphate	77*	79*	80-116

Therefore, the reported results will be qualified as estimated value(“UJ”) for this anion. Percent RPDs were less than 20% for LCS/LCSD.

4.10.4. Field duplicate sample and its associated sample: No field duplicate sample was collected for this sample delivery group.

4.10.5. Raw data was submitted for all requested field samples. Sample TMW48102021 was selected to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met. Due to high concentrations of Sulfate and Chloride, samples were analyzed with high dilution factors on 11-08-21 and 11-09-21 within 28-day holding time.

5.0 CONCLUSION

SDG #21J145 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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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #21J146 Analytical Data Package

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 10-15-21. EMAX Laboratories received the samples on 10-16-21. 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 TMW41102021 (Lab ID#J146-08) was selected to be reviewed as stage 3 deliverable. Raw data for this sample was compared to the reported summary table and went through comprehensive data validation. Sample TMW07102021 (Lab ID#J146-11) was selected by lab to be spiked as MS/MSD. Raw data for this sample together with 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 (13 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 analytical holding times were met for initial and re-analysis of all samples. However, three samples (TMW07102021, TMW15102021 and TMW33102021) were analyzed for Bromide four days past 28-day required holding time. The deviations, if any, are discussed in Section 4.0 for this method.

The SDG # 21J146 analytical data evaluated in this data validation report 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 thirteen (13) water samples were collected on 10-15-21. EMAX Laboratories received the samples on 10-16-21.

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 TMW41102021 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. Sample TMW07102021 was selected to be spiked as MS/MSD by lab. Raw data for this sample together with other QC samples in was also 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 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;
- LCSs 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#21J146 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
TMW01102021	J146-01	10-15-21	S3VM	Anions by IC,
TMW01102021D	J146-02	10-15-21	S3VM	Anions by IC,
TMW02102021	J146-03	10-15-21	S3VM	Anions by IC,
TMW02102021D	J146-04	10-15-21	S3VM	Anions by IC,
TMW55102021	J146-05	10-15-21	S3VM	Anions by IC,
SMW01102021	J146-06	10-15-21	S3VM	Anions by IC,
TMW39S102021	J146-07	10-15-21	S3VM	Anions by IC,
TMW41102021	J146-08	10-15-21	S3VM	Anions by IC,
TMW22102021	J146-09	10-15-21	S3VM	Anions by IC,
TMW23102021	J146-10	10-15-21	S3VM	Anions by IC,
TMW07102021	J146-11	10-15-21	S3VM	Anions by IC,
TMW15102021	J146-12	10-15-21	S3VM	Anions by IC,
TMW33102021	J146-13	10-15-21	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 except for three samples*

*Samples TMW07102021, TMW15102021 and TMW33102021 were analyzed for Bromide four days past 28-day required 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 4.8˚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 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:

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

Completeness (EPA Method 9056A: Anions by IC) =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 thirteen 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: Nitrate-N, Nitrite, Orthophosphate, Bromide, Fluoride Sulfate and Chloride

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 thirteen (13) water samples were collected on 10-15-21. Samples were analyzed on 10-16-21 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Some samples for Bromide, Chloride, fluoride and Sulfate were analyzed and re-analyzed with dilution on 11-09-21, within 28-day holding time. Analysis was all within 28-day holding time. However, samples TMW07042021, TMW15102021 and TMW33102021 were re-analyzed four days past required 28-day holding time for Bromide. Since the exceedance for holding time is less than 2×HT, all positive result for this anion would be qualified as estimated value (“J”) and non-detected results as “UJ” for these samples.

4.1.2. Initial and continuing calibration: Anions such as Bromide, Chloride, Fluoride, 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 on the basis of retention time as compared to reference standards.

Three initial calibration curves were generated with two different instruments with nine calibration levels (0.05-20mg/L) on 08-12-21, 10-07-21 and 11-15-21. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout the analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. 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 08-12-21, 10-07-21 and 11-15-21. Recoveries were all within 90-110% of expected true value. Continuing Calibration standards at 10-injections interval were analyzed on 10-16-21, 11-10-21 and 11-16-21. A total of seven continuing calibration standards were initially analyzed on 10-16-21. Later, ten more continuing calibration standards were analyzed 11-10-21, 11-11-21 and 11-16-21. 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.

4.1.3. Quality Control Samples consisted of two method blanks, two sets of LCS/LCSD, MS/MSD and sample/sample duplicate for initial analysis of all anions. Sample TMW07102021was selected to be spiked as MS/MSD and analyzed as sample/sample duplicate. A third set of MB and LCS/LCD was also analyzed with re-analysis and /or dilutions of samples for Fluoride, Chloride, and Sulfate on 11-10-21 and 11-16-21 (for Bromide). Recoveries of LCS/LCSDs were all within 90-110 % of spiked values for each anion. However, recoveries of Orthophosphate failed the lower acceptance limit in LCS/LCSD analyzed with the second batch. Therefore, the results for associated samples (TMW22102021, TMW23102021, TMW07102021, TMW15102021 and TMW33102021) will be qualified as “UJ” for Orthophosphate.

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

Anion	TMW07102021 MS%	TMW07102021 MSD%	% Acceptance limits
Orthophosphate	43*	45*	80-116
Nitrate	76*	75*	88-111
Bromide	80*	80*	91-110
Chloride	64*	72*	87-111
Fluoride	131*	130*	88-112
Sulfate	118*	97	87-112

*Outside Acceptance limits

Therefore, the reported values for these anions will be qualified as estimated value (“J”) for positive results and as (“UJ”) for non-detect results in the parent sample. Percent RPDs were less than 20% for LCS/LCSD and MS/MSD. Sample TMW07102021 was also used for sample/sample duplicate analysis. All the recoveries and % RPDs were within acceptance limits for each anion.

4.1.4. Field duplicate sample and its associated sample: Sample TMW01102021 was identified as field duplicate of TMW01102021D and sample TMW02102021 was identified as field duplicate of TMW02102021D. Results for each set of sample/sample duplicate is summarized in the tables below:

Anion	TMW01102021 (Lab ID #J146-01) mg/L	TMW01102021D (Lab ID #J146-02) mg/L	**% RPD**
Nitrate	9.5	9.1	4.30
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	1.0	1.0	<1
Chloride	190	190	<1
Fluoride	0.43	0.44	2.30
Sulfate	760	800	5.13

Anion	TMW02102021 (Lab ID #J146-03) mg/L	TMW02102021D (Lab ID #J146-04) mg/L	**% RPD**
Nitrate	81	79	2.5
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.46J	0.49J	6.32
Chloride	310	290	6.66
Fluoride	0.27	0.27	<1
Sulfate	1300	1200	8.00

4.1.5. Raw data was submitted for all requested field samples. Sample TMW41102021 was selected to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met. Samples were analyzed and/or re-analyzed for Bromide, Fluoride, Sulfate and Chloride, on 11-10-21, 11-11-21 within 28-day holding time. However, as mentioned in section 4.1.1, samples TMW07042021, TMW15102021 and TMW33102021 were analyzed four days past required 28-day holding time for Bromide. Since exceedance for holding time is less than 2×HT, the positive results would be qualified as estimated value (“J”) and undetected results as (“UJ) in these samples for Bromide.

5.0 CONCLUSION

SDG #21J146 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 #21J147 Analytical Data Package

Publication Date: 01-27-22

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in October 2021 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 10-14-21. EMAX Laboratories received the samples on 10-16-21. 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 MW38102021(Lab ID# J147-02) was selected 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. Sample BGMW13D102021(Lab ID# J147-10) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with method blank and LCS/LCSD for each method 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, (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 (11 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (9 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (10 samples)
EPA Method SW8332: Nitroglycerine and PETN (10 samples)
EPA Method 8081B: Organochlorine Pesticides (7 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (3 samples)
EPA Method 8151A: Chlorinated herbicides (3 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (7 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (6 samples)
EPA Method 6850: Perchlorate (8 samples)
EPA Method SW6020A: Dissolved and total Metals by ICP-MS (10 samples)
EPA Method 7470A: Mercury & Dissolved Mercury (10 samples)
EPA Method 3520C/8270SIM: 1,4-Dioxane by GC/MS (2 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 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 # 21J147 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 eleven (11) water samples were collected on 10-14-21. EMAX Laboratories received the samples on 10-16-21.

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 MW38102021 (EMAX ID #J147-02) from this sample delivery group was selected as stage 3 data deliverable. Raw data for this sample was evaluated comprehensively. Sample BGMW13D102021 (EMAX ID #J147-10) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with other QC samples (LCS/LCSD) in this report was also 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#21J147 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW47102021	21J147-01	10-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850
MW38102021	21J147-02	10-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls 1,4-Dioxane by 8270 SIM
QC14102021TB9	21J147-03	10-14-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
MW39102021	21J147-04	10-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls 1,4-Dioxane by 8270 SIM

Site Name: Fort Wingate, New Mexico

SDG#21J147 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW32102021	J147-05	10-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
TMW06102021	J147-06	10-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO
TMW06102021D	J147-07	10-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO
TMW24102021	J147-08	10-14-21	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850
TMW03102021	J147-09	10-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#21J147 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
BGMW13D102021	J147-10	10-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
MW22102021	J147-11	10-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Total & Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO
BGMW13D102021MS	J147-10M	10-11-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls
BGMW13D102021MSD	J147-10S	10-04-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury Dissolved Mercury Total Metals by ICP-MS Dissolved Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls

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
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0μg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20μg/L, (Benzidine=40μg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4μg/L
	Nitroglycerine & PETN	SW8332	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	0.5μg/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	None. Holding times were met
Mercury and Dissolved Mercury	Water	Collection to Analysis: 28 days	None. Holding times were met
Anions by IC	Water	Analysis 48 hours from collection For Nitrate, Nitrite and Orthophosphate; 28 days from collection for the rest	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 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 1.4˚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:

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

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:

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

Completeness (EPA Method 5030B/8260C: VOCs) =11/11X100=100%

Completeness (EPA Method 3520B/8270D: SVOCs) =9/9X100=100%

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

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

Completeness (EPA Method 8081B: Organochlorine pesticides) =7/7X100=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) =8/8X100=100%

Completeness (EPA Method 7470A: Mercury and Dissolved Mercury) =10/10X100=100%

Completeness (EPA Method 6020A: Dissolved and Total Metals) =10/10X100=100%

Completeness (EPA Method 3520B/8270SIM: 1,4-Dioxane) =2/2X100=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 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 **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury and Dissolved Mercury by Cold Vapor
EPA Method **3520C/8270** SIM for 1,4-Dioxane by GC/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. Eleven water samples were collected on 10-14-21. Samples were analyzed on 10-25-21 and 10-27-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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 4.1.7.

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 initial calibration on 10-11-21, 10-12-21 and at the beginning of each analysis shift on 10-25-21 and 10-27-21.

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 10-11-21 and 10-12-21. 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 10-11-21	Average Response factor 10-12-21
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.

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) 10-11-21	Least Square Linear Regression (CCF) 10-12-21
2-Chloroethyl vinyl ether	NA	0.9986
1,2-Dibromo-3-chloropropane	NA	0.9995
Vinyl acetate	0.9982	0.9961

Average response factors for all target compounds were within method's recommended values, except for: Acetone (0.050) and 2-Butanone (0.015) in initial calibration analyzed on 10-12-2. 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 10-11-21	Response Factors %RSD 10-12-21
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 10-12-21 and 10-14-21. 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 compound except for Vinyl acetate (30.2% and 25.6%).

Continuing calibration check standards were analyzed at the beginning and end of each analysis shift on 10-25-21 and 10-27-21. Prior to continuing calibration analysis, **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 10-25-21 (I&II)	Continuing cal. Response factors 10-27-21 (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 (10-25-21) I&II	%Deviation from Initial calibration (10-27-21) 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 the initial calibration was less than 20 percent for the rest of target list, except for the compounds listed in the tables below. This should not affect the data quality.

Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-25-21)		%Deviation from Initial calibration (10-27-21)	
		I	II	I	II
2-Chloroethylvinyl ether	≤ 20%	--	23.9*	61.2*	76.5*
4-Methyl-2-Pentanone	≤ 20%	--	--	--	31.7*
Vinyl acetate	≤ 20%	22.3*	--	29.0*	21.3*
2-Butanone	≤ 20%	--	--	--	21.4*

*Outside QC limits

4.1.5. Quality Control samples reported consisted of two method blanks, two sets of LCS/LCSD and MS/MSD. Sample BGMW13D102021was designated to be analyzed 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: Sample TMW06102021 was identified as field duplicate of sample TMW06102021D. Both sample and corresponding field duplicate sample were reported as non-detected for all volatile organic compound list.

4.1.7. Raw data was submitted for all samples. Sample MW38102021 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 nine samples. Water samples were collected on 10-14-21, extracted on 10-21-21 and were analyzed on 10-25-21 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 08-12-21, 08-13-21 and at the beginning of analysis shift on 10-25-21. 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 08-12-21 and 08-13-21. 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 08-12-21
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 08-12-21
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) 08-12-21
Benzoic acid	0.9991
2,4-Dinitrophenol	0.9993
4-Nitrophenol	1.00
4,6-Dinitro-2-methylphenol	0.9983
Benzidine	0.9994
3,3-Dimethylbenzidine	0.9999

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

4.2.4. Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard on 08-12-21 and 08-13-21. 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 except 4,6-Dinitro-2-methylphenol (25.0%). Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 10-25-21. 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 (10-25-21) 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 nearly 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 (10-25-21) 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 listed below in opening and closing continuing calibration standards:

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (10-25-21) I	%Deviation from Initial calibration (10-25-21) II
2,4-Dinitrophenol	≤ 20	23.5*	--
4,6-Dinitro-2-methyl phenol	≤ 20	24.0*	22.0*
3,3-Dimethyl benzidine	≤ 20	21.2*	--

*Exceeded 20% maximum allowed difference in the closing daily standard

Since none of the above compounds were detected in the associated samples and the exceedances were all high biased, this should not affect the quality of data.

4.2.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample BGMW13D102021was 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 within the project acceptance limits for the entire compound list in LCS/LCSD and MS/MSD.

The results and recoveries of LCS/LCSD was used to evaluate accuracy and precision for 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: Sample TMW06102021 was identified as field duplicate of TMW06102021D. Both sample and corresponding field duplicate sample were reported as non-detected for the full Semi-Volatile Compounds list.

4.2.7. Raw data was submitted for all samples. Sample MW38102021 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 seven water samples requested for this method. Water samples were collected on 10-14-21, extracted on 10-20-21 and analyzed on 11-01-21and 11-02-21 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 08-23-21 and before sample analysis on 11-01-21 and 11-02-21. 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 08-23-21. 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 08-14-21 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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 08-14-21 and 08-24-21. 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 five continuing calibration standards were analyzed at 10-injections interval on 11-01-21 and 11-02-21 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, at least three compounds (DDD, DDT, and Endosulfan sulfate) failed the maximum 20% difference in the closing daily standard analyzed on 11-01-21 in column A. Since the recoveries were all high biased and no pesticide was detected in any of the field samples, this should not affect the quality of data. In the five 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 one method blank, one set of LCS/LCSD and MS/MSD. Sample BGMW13D102021was designated to be spiked for MS/MSD. The full pesticide target list was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within established QC limits. Raw data for the QC samples were matching the reported values.

Result for method blank was 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 assigned to this method.

4.3.6. Raw data was submitted for all samples. Sample MW38102021 was selected 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 three water samples requested for this method. Water samples were collected on 10-14-21, extracted on 10-20-21, and analyzed on 10-25-21.

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 10-14-21. 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 10-14-21. 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 would be injected at single point for identification only.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-25-21, 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 both channels.

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, one set of LCS/LCSD and MS/MSD. Sample BGMW13D102021 was designated to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD and MS/MSD.

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 assigned to this method.

4.4.6. Raw data was submitted for all samples. Sample MW38102021 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.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for three water samples requested for this method. Water samples were collected on 10-14-21, extracted on 10-20-21 and analyzed on 11-08-21 and 11-09-21.

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 10-28-21. 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.

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 10-28-21. 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 11-08-21 and 11-09-21, 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 compounds from channels A except Dicamba (23%; column A) and Dinoseb (42%; channel B) in the closing daily standard.

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 BGMW13D102021 was designated to be spiked as MS/MSD. The full 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 sample was assigned to this method.

4.5.6. Raw data was submitted for all samples. Sample MW38102021 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.

4.6. 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 10-14-21, extracted on 10-21-21 and analyzed on 10-25-21 and 10-26-21, 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 08-23-21 and 08-24-21. 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 08-24-21. 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 six continuing calibration standards (both primary and confirmation columns) were analyzed on 10-25-21 and 10-26-21, bracketing the analyses of samples and all the QC samples. Two daily standards were analyzed with Biphenyl column (confirmation column) on 10-26-21. 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 BGMW13D102021 was designated to be spiked as MS/MSD. The full explosive target list was 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 TMW06102021 was identified as field duplicate of sample TMW06102021D. No explosive target compound was detected in each sample and corresponding field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample MW38102021 was selected to be reviewed as stage 3 data deliverable on the chain of custody. Raw data of 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. All positive results, if any were confirmed with confirmation 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 10-14-21, extracted on 10-21-21 and analyzed on 10-21-21.

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 10-21-21, 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 BGMW13D102021was 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 within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** Sample TMW06102021 was identified as field duplicate of sample TMW06102021D. No explosive target compound was detected in each sample and corresponding field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample MW38102021 was selected to be reviewed as stage 3 data deliverable. Raw data of 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. 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 10-14-21. Samples were analyzed on 10-23-21 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 09-09-21. 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 09-09-21. 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 10-23-21, 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 BGMW13D102021 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: Sample TMW06102021 was identified as field duplicate of sample TMW06102021D. No TPH as Gasoline was detected in each sample and corresponding field duplicate sample.

4.8.6. Raw data was submitted for all samples. Sample MW38102021 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 samples requested for this method. Water samples were collected on 10-14-21, extracted on 10-21-21 and analyzed on 10-22-21 within holding time.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 08-12-21. 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 08-12-21. 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 10-22-21 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 BGMW13D102021 was designated to be spiked as MS/MSD. Results and 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: Sample TMW06102021 was identified as field duplicate of sample TMW06102021D. No TPH as Diesel was detected in each sample and corresponding field duplicate sample.

4.9.6. Raw data was submitted for all samples. Sample MW38102021 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 eight water samples requested for this method. Water samples were collected on 10-14-21. Samples were analyzed on 10-19-21 and 10-20-21 within 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 09-21-21. 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 0.999 (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 09-21-21. 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 10-19-21 and 10-20-21, bracketing the analyses and re-analysis 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 BGMW13D102021 was designated to be spiked as MS/MSD. Perchlorate 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: No field duplicate sample was assigned to this method.

4.10.6. Raw data was submitted for all samples. Sample MW38102021 was selected 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 raw data, agreed with all the results reported in data summary reports.

4.11. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.11.1. 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 10-14-21. Samples were prepared (digested) for both total and dissolved metals on 10-21-21. Samples and QC samples were analyzed on 11-12-21 and 11-15-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Three sets of initial calibration curves were established at the start of each analysis day on 11-12-21 (two sets) and 11-15-21. Tune performance report was generated at the start of each analysis day, before initial calibration. It was within method's acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. The correlation coefficient of at least 0.999 was calculated to show the linearity of calibration curve for each metal. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

Metals	Concentration µg/L
Se, Pb, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Mn, Ni, Ag, Tl, V, and Zn	50,100,500,1000
Al, Fe, Ca, Mg, Na, and K	50,000

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of ±20% of the true value for the metals incorporated in each solution.

4.11.3. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample BGMW13D102021 was spiked as MS/MSD. Recoveries of LCS/LCSD were all within the acceptance limit of 80-120% for both total and dissolved metals. Recoveries of MS/MSD were mostly acceptable except for the few as listed in the table below. As a result, all the positive results for parent sample will be qualified as estimated value “J” for these metals. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	BGMW13D102021 **MS%**	BGMW13D102021 **MSD%**	**QC Limit %**	BGMW13D102021 **MS%**	BGMW13D102021 MSD%	**QC Limit %**
Aluminum	√	√	84-117	√	√	84-117
Antimony	√	√	85-117	√	√	85-117
Arsenic	√	√	84-116	√	√	84-116
Barium	87	77*	86-114	110	117*	86-114
Beryllium	√	√	83-121	√	√	83-121
Cadmium	83*	85*	87-115	84*	86*	87-115
Calcium	100	80*	87-118	√	√	87-118
Chromium	√	√	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	√	√	85-118
Iron	√	√	87-118	√	√	87-118
Lead	√	√	88-115	√	√	88-115
Magnesium	√	√	83-118	√	√	83-118
Manganese	√	√	87-115	√	√	87-115
Nickel	√	√	85-117	√	√	85-117
Potassium	√	√	85-115	√	√	85-115
Selenium	√	√	80-120	√	√	80-120
Silver	√	√	85-116	√	√	85-116
Sodium	100	67*	85-117	233*	367*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

Method blank results for metals were acceptable and no contamination was found in the method blanks except for Sodium (120µg/L; >1/2 LOQ) in total metal analysis. Calibration blanks were analyzed after each continuing calibration standard. Sample BGMW13D102021 was used for serial dilution for both total and dissolved metals. Results for serial dilution analysis at 5-fold dilution, was within 10% difference of the initial analysis. The same sample was used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total and dissolved metals.

4.11.4. Field duplicate sample and its associated sample: Sample TMW06102021 was identified as field duplicate of TMW06102021D. Results for each sample/sample duplicate is summarized in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW06102021 (Lab ID#J147-06) µg/L	TMW06102021D (Lab ID#J147-07) µg/L	**% RPD**	TMW06102021 (Lab ID#J147-06) µg/L	TMW06102021D (Lab ID#J147-07) µg/L	**% RPD**
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	0.95J	0.93J	2.13
Barium	14	14	<1	13	13	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	35000	35000	<1	34000	34000	<1
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	U	U	<1	U	U	--
Lead	U	U	--	U	U	--
Magnesium	7700	7800	1.29	73000	72000	1.38
Manganese	39	38	2.60	37	37	<1
Nickel	U	U	--	U	U	--
Potassium	280J	290J	3.51	U	250J	200
Selenium	0.79J	0.82J	3.73	U	U	--
Silver	U	U	--	U	U	--
Sodium	900000	900000	<1	910000	910000	<1
Thallium	U	U	--	U	U	--
Vanadium	3.6J	4.0J	10.5	3.6J	3.7J	2.74
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Sample MW38102021 was selected to be reviewed as stage 3 data deliverable on the chain of custody. Raw data of 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.

4.12. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.12.1. 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 10-14-21. Samples were prepared (digested) and analyzed for Mercury and dissolved Mercury on 10-27-21. All samples were preserved and filtered for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 10-27-21. The correlation coefficient of at least 0.999911 was calculated to show the linearity of calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L. Initial calibration verification and Continuing calibration verification standards for mercury was within the acceptable range (90-110% of the spiked value).

4.12.3. Quality Control samples consisted of method blank one set of LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Sample BGMW13D102021 was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample BGMW13D102021 was also used for serial dilution for Mercury and dissolved Mercury. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in each method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample TMW06102021 was identified as field duplicate of TMW06102021D. No Mercury was detected in each of the sample and corresponding field duplicate sample for Mercury and dissolved Mercury.

4.12.5. Raw data was submitted for all samples. Sample MW38102021 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 raw data, agreed with all the results reported in data summary reports.

4.13. 1,4-Dioxane (EPA Method 3520C/8270SIM)

4.13.1. Technical Holding Times

Holding time requirement was met for two samples requested for this method. Water samples were collected on 10-14-21, extracted on 10-21-21 and were analyzed on 10-22-21; within holding time.

Selective Ion Monitoring (SIM) method with GC/MS was used for analysis. Isotopically-labeled 1,4-Dioxane-d8 was added to serve as internal standard.

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 4.14.7.

4.13.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 08-04-21 and at the beginning of analysis batch on 10-22-21. It passed all the method assigned criteria.

4.13.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD.

Initial calibration curve was generated on 08-04-21. A multi-level calibration standard ranging from 0.05mg/L to 50mg/L was used for this purpose. Internal standard curve type in Selective Ion Monitoring (SIM) mode was used for initial calibration and all following analysis. 1,4-Dioxane-d8 was used as internal standard. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table. Average response curve type was used for 1,4-Dioxane and Bromobenzene as surrogate. Target compounds met the maximum 15% RSD limit (all less than 15%).

4.13.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 08-04-21. Percent difference (%D) were within ±20% of the expected value.

Continuing calibration check standard was analyzed at the beginning and at the end of analysis shift on 10-22-21. Prior to opening continuing calibration standard, instrument performance tune check standard (BFB) was carried out. It passed all the method tuning criteria. Percent difference (%D) and/or drift for both opening and closing continuing calibration standards were within ±20% of the expected value for each daily standard.

4.13.5. Quality Control: The QC samples reported consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. 1,4-Dioxane as the only target with Bromobenzene as surrogate was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits.

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 method’s acceptance limit.

4.13.6. **Field duplicate sample** and its associated sample: No field duplicate sample was assigned for this method.

4.13.7. Sample MW38102021 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 #21J147 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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