Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22D091 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 April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of eight (8) water samples were collected on 04-11-22. EMAX Laboratories received the samples on 04-12-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples was submitted for the requested analytical method. Sample BGMW07042022 (Lab ID#D091-01) was selected to be reviewed as stage 3 deliverable. Raw data for this sample was compared to the reported summary tables and went through comprehensive data validation. No sample was designated to be spiked as MS/MSD. Recoveries and results of LCS/LCSD was used to evaluate accuracy and precision. Raw data for method blanks and LCS/LCSDs were also cross checked with the corresponding summary table results.

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

EPA Method 9056A: Anions by IC (8 samples)

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

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

The SDG # 22D091 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 eight (8) water samples were collected on 04-11-22. EMAX Laboratories received the samples on 04-12-22.

2.1 Data Reporting

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

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)
- Result and percent recoveries of MS/MSD, 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 BGMW07042022 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample together with QC samples were evaluated comprehensively at stage 2b and stage 3 data validation review. No sample was designated to be spiked as MS/MSD on the chain of custody. Raw data each set of LCS/LCSD was also reviewed. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);
- Technical holding times were confirmed for all samples with 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;
- LCS/LCSD results were evaluated; and
- Method blank results as well as surrogate recoveries were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#22D091 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
BGMW07042022	D091-01	04-11-22	S3VM	Anions by IC,
MW23042022	D091-02	04-11-22	S3VM	Anions by IC,
MW32042022	D091-03	04-11-22	S3VM	Anions by IC,
BGMW03042022	D091-04	04-11-22	S3VM	Anions by IC,
BGMW12042022	D091-05	04-11-22	S3VM	Anions by IC,
TMW18042022	D091-06	04-11-22	S3VM	Anions by IC,
BGMW08042022	D091-07	04-11-22	S3VM	Anions by IC,
TMW36042022	D091-08	04-11-22	S3VM	Anions by IC,

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

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

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

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

TABLE 2-2
Summary of Analytical Methods and Holding Time Requirements
USACE Wingate, New Mexico

ANALYSIS Method	MATRIX	HOLDING TIME REQUIREMENT	DATA QUALIFIED AS "J"
Anions by IC	Water	Analysis within 48 hours for Nitrate, Nitrite and Orthophosphate	None. Holding times were met
		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 2.7˚C upon arrival. Samples were received in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

Precision is a measure of the closeness with which multiple analyses of a given sample agree with each other. It describes the agreement between two or more measurements that have been made in 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 9056A: Anions) =8/8X100=100%

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

4.0 DATA VALIDATION

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

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

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

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

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

The following Reason codes were applied in the report:

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

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

4.1.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of water samples requested for this method. A total of eight (8) water samples were collected on 04-11-22. Samples were analyzed on 04-12-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Some samples were analyzed on 04-18-22 and 04-20-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

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

Instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-04-22. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each anion. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of initial calibration on 03-04-22. Recoveries were all within

90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 04-12-22, 04-18-22 and 04-20-22. A total of seven continuing calibration standards were analyzed with analysis and re-analysis of all samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.12.3. Quality Control Samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Two sets of LCS/LCSD were analyzed with Chloride and three sets of LCS/LCSD were analyzed with Sulfate analysis. Recoveries of LCS/LCSDs were within 90-110 % of spiked values for each anion.

Percent RPDs were less than 20% for each set of LCS/LCSD for all anions.

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

4.12.5. Raw data was submitted for all requested field samples. Sample BGMW07042022 (Lab ID#22D091-01) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #22D091 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.

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USACE Fort Wingate Depot Activity
New Mexico

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5.0 CONCLUSION 43

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of eleven (11) water samples were collected on 04-11-22. EMAX Laboratories received the samples on 04-13-22. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample MW32042022(Lab ID# D109-05) 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 review. No sample was designated to be spiked as MS/MSD on the chain of custody, therefore the results and recoveries of LCS/LCSD was used for both accuracy and precision. Raw data for method blank and LCS/LCSD for each method were cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (11 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (10 samples)
EPA Method SW846 3535A/8330B: Nitroaromatics and Nitramines (10 samples)
EPA Method 8081B: Organochlorine Pesticides (7 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 (10 samples)
EPA Method SW6020A: Dissolved and total Metals by ICP-MS (10 samples)
EPA Method 7470A: Mercury & Dissolved Mercury (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 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 # 22D109 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. Method SW846 3535A/8330B (Nitroaromatics and Nitramines) and method SW846 6850 (Perchlorate) was sub contracted by EMAX to GEL Laboratories in South Carolina.

A total of eleven (11) water samples were collected on 04-11-22. EMAX Laboratories received the samples on 04-13-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the data was subjected to validation to the equivalent of stage 3.

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if submitted

- Results and percent recoveries of Lab Control Samples (LCS/LCSD)
- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses and chromatograms for the sample/samples at Stage 3 deliverable and related QC samples.

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. One field sample MW32042022 (EMAX ID #D109-05) 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#22D109 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
BGMW07042022	22D109-01	04-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Modified
MW23042022	22D109-02	04-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified
QC11042022TB1	22D109-03	04-11-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW16042022	22D109-04	04-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified
MW32042022	22D109-05	04-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Modified

Site Name: Fort Wingate, New Mexico

SDG#22D109 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
BGMW12042022	22D109-06	04-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Perchlorate by 6850 Modified
BGMW03042022	22D109-07	04-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Organochlorine Pesticides
TMW19042022	22D109-08	04-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
BGMW08042022	22D109-09	04-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls

Site Name: Fort Wingate, New Mexico

SDG#22D109 **Matrix: Water**

Field/Client ID	Lab ID	Date collected	Validation stage	Requested Methods of Analysis
TMW18042022	22D109-10	04-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
TMW36042022	22D109-11	04-11-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines 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	SW3535A/8330B	1.25-5µg/L
	Nitroglycerine & PETN	SW3535A/8330B	2.5µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in 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
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 eleven ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 0.8˚C and as high as 4.8˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

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

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

Where:

RPD = Relative percent difference

R_1 = Result of the first duplicate or measured sample concentration

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

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

3.2.2 Accuracy

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

3.2.3 Completeness

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

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

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

Completeness (EPA Method 5030B/8260C: VOCs) =11/11X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =10/10X100=100%
Completeness (EPA Method 3535A/8330B: Explosives) =10/10X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =7/7X100=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) =10/10X100=100%
Completeness (EPA Method 7470A: Mercury & Dissolved Mercury) =10/10X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =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 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 **3535A/8330B** for Nitrtoaromatics and Nitramine by LC/MS/MS
EPA Method **6850** for Perchlorate by HPLC/MS/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury & 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 eleven samples. Water samples were collected on 04-11-22. Samples were analyzed on 04-25-22 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 04-05-22 and at the beginning of analysis shift on 04-25-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-05-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-05-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.022), 2-Butanone (0.010), 2-Hexanone (0.062) and 4-Methyl-2-pentanone (0.086). 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 04-05-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-06-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standard was analyzed at the beginning and end of analysis shift on 04-25-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-25-22 (I)	Continuing cal. Response factors 04-25-22 (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 (04-25-22) I	%Deviation from Initial calibration (04-25-22) 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 1,2-Dichloroethane (27.9%) and Dibromoethane (23.3%) in the closing daily standard. 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 MW32042022 (Lab ID# 22D109-05) 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 04-11-22, extracted on 04-13-22 and were analyzed on 04-18-22 within required holding time.

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

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

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 04-18-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

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

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

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

√ denotes passing method acceptance limits

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

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

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

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

√ denotes passing method acceptance limits

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

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

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

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

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-18-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

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

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (04-18-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (04-18-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list.

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

Compound	LCS	LCSD	**QC Limit%**
2,4-Dichlorophenol	46%*	57%	47-121
2-Chlorophenol	34%*	44%	38-117
2-Nitrophenol	42%*	52%	47-123
Phenol	42%*	51%	50-130
2,6-Dichlorophenol	46%*	56%	50-118

*Outside QC limit

Results and recovery of LCSD was used to evaluate the QC for these compounds.
Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision for the rest of target list in this method.

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

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

4.2.7. Raw data was submitted for all samples. Sample MW32042022(Lab ID#22D109-05) 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 seven water samples requested for this method. Water samples were collected on 04-11-22, extracted on 04-14-22 and analyzed on 04-18-22 and 04-20-22 within required holding time.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 03-11-22, 03-12-22 and before sample analysis on 04-18-22 and 04-20-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

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

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

4.3.3. Calibration Verification and Continuing Calibration

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

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

A total of six continuing calibration standards were analyzed at 10-injections interval on 04-18-22 and 04-20-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. However, %D for all pesticide target list failed the maximum 20% difference (all high biased) for channel B in three daily standards analyzed on 04-20-22. Therefore, corresponding samples were evaluated with channel A only. In the six continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

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

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

Surrogate recoveries were all within the method QC acceptance limits.

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

4.3.6. Raw data was submitted for all samples. Sample MW32042022(Lab ID#22D109-05) 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 04-11-22, extracted on 04-14-22, and analyzed on 04-15-22 and 04-19-22, within holding time.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-17-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-17-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-15-22 and 04-19-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

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

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD, 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 MW32042022(Lab ID#22D109-05) 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 04-11-22, extracted on 04-15-22 and analyzed on 04-20-22.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 04-19-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99776 was used for MCPP in column B.

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

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 04-19-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-20-22 and 04-21-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels.

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

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

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

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

4.5.6. Raw data was submitted for all samples. Sample MW32042022 (Lab ID#22D109-05) 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 LC/MS/MS (EPA Method3535A/ 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 04-11-22, extracted on 04-18-22 and analyzed on 04-25-22 and 04-28-22, within holding time.

A High-Performance LC (HPLC) coupled with two Mass detectors in tandem (LC/MS/MS) was used for analysis. Samples were sub-contracted to GEL Laboratories in South Carolina by EMAX Laboratory.

4.6.2. Initial Calibration

Two initial calibration curves were performed with seven concentration levels for each analyte on 04-25-22 and 04-27-22. Internal standard curve type was used for initial calibration and all the following analysis. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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.)

4.6.3. Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard (ICV) for all target analytes on 04-25-22 and 04-28-22. Percent recoveries were within required method limits (70-130% of the true value).

Continuing calibration standards were analyzed at the start of analysis on 04-25-22 and 04-28-22. Percent recoveries were within required method limits (80-120% of the true value) for all target compounds except HMX (139%) and RDX (135%) in the daily standard analyzed on 04-28-22. Since the recoveries were biased high and target analytes were not detected in the associated samples, it should not affect the data quality. Initial calibration blanks and continuing calibration blanks were all non-detect.

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. 3,4-Dinitrotoluene was used as surrogate.

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 MW32042022 was 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.

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

4.7.1. 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 04-11-22. Samples were analyzed on 04-15-22 within holding time requirement.

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

4.7.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-07-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-15-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.7.4. 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. 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.

4.7.5. 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 MW32042022(Lab ID#22D109-05) 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.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for two water sample requested for this method. Water samples were collected on 04-11-22, extracted on 04-14-22 and analyzed on 04-18-22.

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

4.8.2. 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.

4.8.3. 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 04-18-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated 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.

4.8.5. 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 MW32042022(Lab ID#22D109-05) 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. Perchlorate by HPLC/MS/MS (EPA Method 6850 Modified)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for ten samples requested for this method. Water samples were collected on 04-11-22. Samples were prepared on 04-29-22 and analyzed on 05-03-22.

A High-Performance LC coupled with two Mass Detectors (HPLC/MS/MS) was used for analysis. The sample for this method was sub-contracted by EMAX Laboratory to GEL (General Engineering Laboratories) in South Carolina.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-30-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^-$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.99993 (Perchlorate) and 0.99992 (perchlorate 101) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.05 – 10 µg/L.

Retention time for each isotope at each calibration level was within 0.2 minutes required by the method.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-30-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of seven daily standards were carried out on 05-03-22 and 05-04-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.9.4. Quality Control samples consisted of one method blank, one LCS and MS/MSD. Sample BGMW07042022 was selected to be spiked as MS/MSD. Perchlorate was spiked and reported for LCS and MS/MSD. Percent recoveries (%R) were within the established QC limits LCS and MS/MSD (84-119%). Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Interference check sample (ICS) was analyzed and results were within acceptable limit (80-120% of the true value).

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.

4.9.5. 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 MW32042022 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. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.10.1. 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 04-11-22. Samples were prepared (digested) for both total and dissolved metals on 04-15-22. Samples and QC samples were analyzed on 04-18-22 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.

4.10.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 04-18-22. 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. Five levels of concentration were used for initial calibration. 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.

4.10.3. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD for total metals. Sample BGMW07042022 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 summarized in the table below. As a result, all the positive results will be qualified as estimated value "J" for these metals in the parent sample for total metals. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	BGMW07042022 MS%	BGMW07042022 MSD%	**QC Limit %**	NA	NA	**QC Limit%**
Aluminum	111	121*	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	433*	-67*	87-118	NA	NA	87-118
Chromium	111	127*	85-116	NA	NA	85-116
Cobalt	√	√	86-115	NA	NA	86-115
Copper	√	√	85-118	NA	NA	85-118
Iron	√	√	87-118	NA	NA	87-118
Lead	√	√	88-115	NA	NA	88-115
Magnesium	30*	17*	83-118	NA	NA	83-118
Manganese	100	33*	87-115	NA	NA	87-115
Nickel	122*	126*	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	-3000*	-1000*	85-117	NA	NA	85-117
Thallium	√	√	82-116	NA	NA	82-116
Vanadium	83*	84*	86-115	NA	NA	86-115
Zinc	0.00*	0.00*	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 BGMW07042022 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.

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

4.10.5. Raw data was submitted for all samples. Sample MW32042022 (Lab ID#22D109-05) 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.

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

4.11.1. 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 04-11-22. Samples were prepared (digested) and analyzed for Mercury and dissolved Mercury on 04-18-22. All samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.11.2. 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 04-18-22. Correlation coefficient of at least 0.999327 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).

4.11.3. Quality Control: The data for all the QC samples were within acceptable control limits. QC samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW16042022 was selected to be spiked as MS/MSD Mercury and sample MW23042022 was spiked as MS/MSD for dissolved Mercury. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for both Mercury and dissolved Mercury. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

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

4.11.5. Raw data was submitted for all samples. Samples MW32042022 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 #22D109 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
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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of fourteen (14) water samples were collected on 04-12-22. EMAX Laboratories received the samples on 04-13-22. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Two samples from this sample delivery group, TMW44042022(Lab ID#D120-05) and TMW46042022 (Lab ID# D120-07) were designated as stage 3 deliverable. Raw data for these samples was compared to the reported summary tables for each method and went through comprehensive data validation review. No sample was designated to be spiked as MS/MSD on the chain of custody, therefore the results and recoveries of LCS/LCSD was used for both accuracy and precision. Raw data for method blank and LCS/LCSD for each method were cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (14 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (11 samples)
EPA Method SW846 3535A/8330B: Nitroaromatics and Nitramines (13 samples)
EPA Method 8081B: Organochlorine Pesticides (12 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (5 sample)
EPA Method 8151A: Chlorinated herbicides (5 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (6 samples)

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

EPA Method 6850: Perchlorate (13 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (13 samples)

EPA Method 7470A: Mercury & Dissolved Mercury (13 samples)
EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate (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 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 # 22D120 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. Method SW3535A/8330B (Nitroaromatics and Nitramines) and method SW846 6850 (Perchlorate) was sub contracted by EMAX to GEL Laboratories in South Carolina.

A total of fourteen (14) water samples were collected on 04-12-22. EMAX Laboratories received the samples on 04-13-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the data was subjected to validation to the equivalent of stage 3.

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if submitted

- Results and percent recoveries of Lab Control Samples (LCS/LCSD)
- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses and chromatograms for the sample/samples at Stage 3 deliverable and related QC samples.

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. Two field samples TMW44042022 (EMAX ID #D120-05) and TMW46042022 (EMAX ID#D120-07) from this sample delivery group were 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#22D120 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW58042022	22D120-01	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Modified TPH Gasoline; TPH as DRO Anions by IC
BGMW09042022	22D120-02	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Modified Anions by IC
BGMW09042022D	22D120-03	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Modified Anions by IC
TMW37042022	22D120-04	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22D120 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW44042020	22D120-05	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified Anions by IC
BGMW10042022	22D120-06	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Organochlorine Pesticides Anions by IC
TMW46042022	22D120-07	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Organochlorine Pesticides Anions by IC
MW24042022	22D120-08	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines 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#22D120 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW01042022	22D120-09	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Mercury & Dissolved Mercury Dissolved Metals by ICP- MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Anions by IC Perchlorate by 6850
MW24042022D	22D120-10	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Mercury & Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
MW02042022	22D120-11	04-12-22	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Mercury & Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Anions by IC
QC12042022EB1	22D120-12	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Mercury & Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Modified TPH Gasoline; TPH as DRO Anions by IC
SMW01042022	22D120-13	04-12-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Mercury & Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Anions by IC
QC12042022TB2	22D120-14	04-12-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

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

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 μg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20μg/L, (Benzidine=40μg/L)
	Nitroaromatics &Nitramines	SW3535A/8330B	1.25-5μg/L
	Nitroglycerine & PETN	SW3535A/8330B	2.5μg/L
	Chlorinated Herbicides	SW8151A	1μg/L, (MCPA=40μg/L)
	Organochlorine Pesticides	SW8081B	0.1μg/L Methoxychlor =1.0μg/L Toxaphene =2.0μg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1μg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100μg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5μg/L,1μg/L,20μg/L,100μg/L,200μg/L
	Dissolved Mercury/Mercury	SW7470A	0.5μg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2μg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements with qualifications if applied.

TABLE 2-2
Summary of Analytical Methods and Holding Time Requirements
USACE Wingate, New Mexico

ANALYSIS Method	MATRIX	HOLDING TIME REQUIREMENT	DATA QUALIFIED AS "J"
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	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:

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

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) =14/14X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =11/11X100=100%
Completeness (EPA Method 3535A/8330B: Explosives) =13/13X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =12/12X100=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) 6/6X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =5/5X100=100%
Completeness (EPA Method 6850: Perchlorate) =13/13X100=100%
Completeness (EPA Method 7470A: Mercury & Dissolved Mercury) =13/13X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =13/13X100=100%
Completeness (EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate) =13/13X100=100%

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

4.0 DATA VALIDATION

This data review covers fourteen water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **3535A/8330B** for Nitrtoaromatics and Nitramine by LC/MS/MS
EPA Method **6850** for Perchlorate by HPLC/MS/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 fourteen samples. Water samples were collected on 04-12-22. Samples were analyzed on 04-25-22 and 04-26-22 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

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-21-22 and at the beginning of each analysis shift on 04-25-22 and 04-26-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-21-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-21-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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) 04-21-22
Vinyl Chloride	0.9988
Acetone	0.9984
Tetrahydrofuran	0.9972

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.049) and 2-Butanone (0.017). 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 04-21-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-22-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standard was analyzed at the beginning and end of each analysis shift on 04-25-22 and 04-26-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-25-22 (I &II)	Continuing cal. Response factors 04-26-22 (I&II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-25-22) I&II	%Deviation from Initial calibration (04-25-22) I&II
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for target analytes listed in the table below for each opening and closing daily standards. This should not affect the data quality.

Target analyte	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-25-22)		%Deviation from Initial calibration (04-26-22)	
		I	II	I	II
Vinyl chloride	≤ 20%	24%*	--	--	--
2-Chloroethyvinylether	≤ 20%	22%*	63.4%*	38.2%*	25.7%*
Chloromethane	≤ 20%	--	41.4%*	--	--
2,2-Dichloropropane	≤ 20%	--	24.7%*	--	--
Acetone	≤ 20%	--	--	22.4%*	--

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

4.1.6. **Field duplicate sample** and its associated sample: Field sample BGMW09042022 was identified as field duplicate of sample BGMW09042022D and field sample MW24042022 was identified as field duplicate of sample MW24042022D. No VOCs was detected in any of field sample or associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Samples TMW44042022 (Lab ID# 22D120-05) and TMW46042022 (Lab ID# 22D120-07) were designated to be reviewed as stage 3 data deliverable. Raw data for these samples with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

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

4.2.1. Technical Holding Times

Holding time requirement was met for all eleven samples. Water samples were collected on 04-12-22, extracted on 04-18-22 and were analyzed on 04-21-22 within required holding time.

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

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

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 04-21-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

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

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

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

√ denotes passing method acceptance limits

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

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

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

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

√ denotes passing method acceptance limits

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

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

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

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

4.2.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-21-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

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

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	**Minimum response factor (Method limits)**	**Continuing cal. Response factors (04-21-22) I&II**
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for 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 (04-21-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
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:

Target Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (04-21-22) I	%Deviation from Initial calibration (04-21-22) II
Nitrophenol	≤ 20	22.0%*	--
Benzoic acid	≤ 20	--	20.5%*

*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 mostly within the project acceptance limits, except for two compounds in LCS as listed in the table below:

Target analyte	LCS	LCSD	**QC Limit%**
2,6-Dichlorophenol	49%*	55%	50-118
Phenol	47%*	50%	50-130

*Outside QC limit

Results and recoveries of LCSD was used to evaluate the QC for these compounds.
Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision for the rest of compound list in this method.

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

4.2.6. **Field duplicate sample** and its associated sample: Field sample BGMW09042022 was identified as field duplicate of sample BGMW09042022D and field sample MW24042022 was identified as field duplicate of sample MW24042022D. No SVOCs was detected in any of field sample or associated field duplicate sample.

4.2.7. Raw data was submitted for all samples. Samples TMW44042022 (Lab ID# 22D120-05) and TMW46042022 (Lab ID# 22D120-07) 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.

4.3. 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 twelve water samples requested for this method. Water samples were collected on 04-12-22, extracted on 04-14-22 and analyzed on 04-18-22, 04-19-22 and 04-20-22 within required holding time.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 03-11-22, 03-12-22 and before sample analysis on 04-18-22, 04-19-22 and 04-20-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

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

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

4.3.3. Initial Calibration Verification and Continuing Calibration

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

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

A total of seven continuing calibration standards were analyzed at 10-injections interval on 04-18-22, 04-19-22 and 04-20-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. However, %D for all pesticide target list failed the maximum 20% difference (all high biased) for channel B in three daily standards analyzed on 04-20-22. Therefore, corresponding samples were evaluated with channel A only. In all continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

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

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

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Field sample BGMW09042022 was identified as field duplicate of sample BGMW09042022D and field sample MW24042022 was identified as field duplicate of sample MW24042022. No organochlorine pesticide was detected in any of field samples or associated field duplicate samples.

4.3.6. Raw data was submitted for all samples. Samples TMW44042022 (Lab ID# 22D120-05) and TMW46042022 (Lab ID# 22D120-07) 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 water samples requested for this method. Five water samples were collected on 04-12-22, extracted on 04-14-22, and analyzed on 04-15-22 and 04-19-22.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-17-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-17-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-15-22 and 04-19-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

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

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD, 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: Field sample BGMW09042022 was identified as field duplicate of sample BGMW09042022D. No Polychlorinated Biphenyls (PCBs) was detected in any of field sample or associated field duplicate sample.

4.4.6. Raw data was submitted for all samples. Sample BGMW10042022(Lab ID#22D120-06) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Five water samples were collected on 04-12-22, extracted on 04-15-22 and analyzed on 04-20-22.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 04-19-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99776 was used for MCPP in column B.

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

4.5.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 04-19-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-20-22 and 04-21-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels.

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

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

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

4.5.5. Field duplicate sample and its associated sample: Field sample BGMW09042022 was identified as field duplicate of sample BGMW09042022D. No Chlorinated Herbicide was detected in the field sample or associated field duplicate sample.

4.5.6. Raw data was submitted for all samples. Sample BGMW10042022 (Lab ID#22D120-06) 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 LC/MS/MS (EPA Method3535A/ 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen (13) water samples requested for this method. Water samples were collected on 04-12-22, extracted on 04-19-22 and analyzed on 04-26-22, 05-04-22 and 05-09-22, within holding time.

A High-Performance LC (HPLC) coupled with two Mass Detectors in tandem (LC/MS/MS) was used for analysis. Samples were sub-contracted to GEL Laboratories in South Carolina by EMAX Laboratory.

4.6.2. Initial Calibration

Two different instruments were calibrated and used for analysis. Four initial calibration curves were performed with seven concentration levels for each analyte on 04-25-22, 04-27-22, 05-03-22 and 05-08-22. Internal standard curve type was used for initial calibration and all the following analysis. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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) for all target analytes.

4.6.3. Initial Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard (ICV) for each target analyte on 04-25-22, 04-28-22, 05-04-22 and 05-08-22. Percent recoveries were within required method limits (70-130% of the true value).

Continuing calibration standards were analyzed at the start and end of each analysis shift on 04-26-22, 05-04-22 and 05-09-22. Percent recoveries were within required method limits (80-120% of the true value) for all target compounds except Nitrobenzene (122%), o-Nitrotoluene (127%) and p-Nitrotoluene (128%) in the daily standard analyzed on 05-09-22. Four last samples were associated with this standard. Since the recoveries were biased high and target analytes were not detected in the associated samples, it should not affect the data quality. Initial calibration blanks and continuing calibration blanks were all non-detect.

4.6.4. Quality Control samples consisted of one method blank and one of LCS only. No sample was designated to be spiked as MS/MSD. The entire explosive target lists were spiked and reported for LCS. 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. 3,4-Dinitrotoluene was used as surrogate.

4.6.5 Field duplicate sample and its associated sample**:** Field sample BGMW09042022 was identified as field duplicate of sample BGMW09042022D and field sample MW24042022 was identified as field duplicate of sample MW24042022D. No explosives were detected in any of the field samples or associated field duplicate samples.

4.6.6. Raw data was submitted for all samples. Samples TMW44042022(Lab ID#22D120-05) and TMW46042022(Lab ID#22D120-07) were designated as stage 3 data deliverable. Raw data foe 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.

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

4.7.1. 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 04-12-22. Samples were analyzed on 04-14-22 and 04-15-22 within holding time requirement.

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

4.7.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-07-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.7.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-14-22 and 04-15-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.7.4. 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. 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.

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

4.7.6. Raw data was submitted for all samples. Sample TMW46042022(Lab ID#22D120-07) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.8.1. 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 04-12-22, extracted on 04-14-22 and analyzed on 04-18-22, 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.

4.8.2. 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.

4.8.3. Initial 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%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-18-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated 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.

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

4.8.6. Raw data was submitted for all samples. Sample TMW46042022(Lab ID#22D120-07) 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. Perchlorate by HPLC/MS/MS (EPA Method 6850 Modified)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen samples requested for this method. Water samples were collected on 04-12-22. Samples were prepared on 05-02-22 and analyzed on 05-04-22.

A High-Performance LC coupled with two Mass Detectors (HPLC/MS/MS) was used for analysis. The sample for this method was sub-contracted by EMAX Laboratory to GEL (General Engineering Laboratories) in South Carolina.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 05-04-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.99985 (Perchlorate) and 0.99979 (perchlorate 101) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.05 – 10 µg/L.

Retention time for each isotope at each calibration level was within 0.2 minutes required by the method.

4.9.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 05-04-22. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of three daily standards were carried out on 05-04-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 90-110% limit.

4.9.4. Quality Control samples consisted of one method blank, one LCS and MS/MSD. Sample TMW58042022 was selected to be spiked as MS/MSD. Perchlorate was spiked and reported for LCS and MS/MSD. Percent recoveries (%R) were within the established QC limits LCS and MS/MSD (84-119%). Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Interference check sample (ICS) was analyzed and results were within acceptable limit (80-120% of the true value).

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.

4.9.5. Field duplicate sample and its associated sample: Field sample BGMW09042022 was identified as field duplicate of sample BGMW09042022D and field sample MW24042022 was identified as field duplicate of sample MW24042022D. No Perchlorate was detected in any of the field samples or associated field duplicate samples.

4.10.6. Raw data was submitted for all samples. Sample TMW44042022 (Lab ID#22D120-05) and TMW46042022 (Lab ID#22D120-07) 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.

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

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen water samples requested for this method. Water samples were collected on 04-12-22. Samples were prepared (digested) for both total and dissolved metals on 04-15-22. Samples and QC samples were analyzed on 04-22-22 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.

4.10.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 04-22-22. 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. Five levels of concentration were used for initial calibration. 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.

4.10.3. Quality Control samples consisted of 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.

4.10.4. Field duplicate sample and its associated sample: Field sample BGMW09042022 was identified as field duplicate of sample BGMW09042022D and field sample MW24042022 was identified as field duplicate of sample MW24042022D. Results and %RPD for each set of field sample and associated field duplicate sample are listed in the tables below:

ANALYTE	Total Metals			Dissolved Metals		
	BGMW09042022 µg/L	BGMW09042022D µg/L	% RPD	BGMW09042022 µg/L	BGMW09042022D µg/L	% RPD
Aluminum	740J	730J		U	U	--
Antimony	U	U	--	U	U	--
Arsenic	1.4J	1.3J	7.41	1.3J	1.3J	<1
Barium	11	14		11	9.8	
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	36000	36000	<1	34000	34000	<1
Chromium	2.7J	2.9J		U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	480J	500J		U	U	--
Lead	U	0.84J	200	U	U	--
Magnesium	4200	4200	<1	4000	4100	
Manganese	98	120		15	13	
Nickel	4.3J	4.8J		U	U	--
Potassium	1600J	1600J	<1	1500J	1500J	<1
Selenium	78	76	2.60	U	U	--
Silver	U	U	--	U	U	--
Sodium	1200000	1200000	<1	1200000	1200000	<1
Thallium	U	U	--	U	U	--
Vanadium	6.5J	6.1J		6.9J	7.5J	
Zinc	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	MW240420222 µg/L	MW240420222D µg/L	% RPD	MW240420222 µg/L	MW240420222D µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	0.86J	0.83J		0.91J	0.69J	

ANALYTE	Total Metals			Dissolved Metals		
	MW240420222 µg/L	MW240420222D µg/L	% RPD	MW240420222 µg/L	MW240420222D µg/L	% RPD
Barium	300	280		200	210	
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	34000	33000		33000	33000	<1
Chromium	0.61J	0.67J		U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	2000	1900		U	170J	200
Lead	U	U	--	U	U	--
Magnesium	11000	11000	<1	11000	11000	<1
Manganese	470	470	<1	440	440	<1
Nickel	U	U	--	U	U	--
Potassium	820J	800J		820J	810J	
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	270000	270000	<1	270000	270000	<1
Thallium	U	U	--	U	U	--
Vanadium	1.3J	1.9J		2.8J	U	200
Zinc	U	U	--	U	U	--

4.10.5. Raw data was submitted for all samples. Sample TMW44042022 (Lab ID#22D120-05) and TMW46042022 (Lab ID#22D120-07) 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. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen (13) water samples requested for this method. Water samples were collected on 04-12-22. Samples were prepared (digested) on 04-20-22 (for Mercury) and 04-22-22 (for dissolved Mercury). Samples were analyzed for Mercury on 04-21-22 and for dissolved Mercury on 04-22-22. All samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.11.2. Initial and continuing calibrations: The instrument calibrations for this method were acceptable. One blank and five standard levels were used for calibration curve at the beginning of each analysis day on 04-21-22 and 04-22-22. The correlation coefficient of at least 0.999275 and 0.999821 was calculated to show the linearity of each calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

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

4.11.4. Field duplicate sample and its associated sample: Field sample BGMW09042022 was identified as field duplicate of sample BGMW09042022D and field sample MW24042022 was identified as field duplicate of sample MW24042022D. No Mercury was detected in each set of sample and associated field duplicate sample.

4.11.5. Raw data was submitted for all samples. Sample TMW44042022 (Lab ID#22D120-05) and TMW46042022 (Lab ID#22D120-07) 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.

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

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

04-12-22. Samples were analyzed on 04-13-22 and 04-14-22, within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Some samples were re-analyzed at higher dilutions on 04-25-22, 04-26-22 and 05-05-22 for Chloride and Sulfate within 28-day holding times.

4.12.2. Initial and continuing calibration: Anions such as Chloride, Fluoride, Bromide, Nitrite, Nitrate, Orthophosphate and Sulfate were separated from water samples by Ion chromatography. The separated anions in their acid form (very conductive) were measured by conductivity. They were identified 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 03-04-22 and 05-04-22. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each anion. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 03-04-22 and 05-04-22. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 04-13-22, 04-14-22, 04-25-22, 04-26-22 and 05-05-22. A total of fourteen continuing calibration standards were analyzed with analysis and re-analysis of all samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.12.3. Quality Control Samples consisted of two method blanks and two sets of LCS/LCSD. No sample was designated to be spiked as MS/MSD. A third and fourth sets of QC samples (MB and LCS/LCD) was analyzed with dilutions and/or re-analysis of samples for Chloride 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.

4.12.4. Field duplicate sample and its associated sample: Field sample BGMW09042022 was identified as field duplicate of sample BGMW09042022D and field sample MW24042022 was identified as field duplicate of sample MW24042022D. Results for sample/sample duplicates are summarized in the table below:

Anion	BGMW09042022 (Lab ID #D120-02) mg/L	BGMW09042022D (Lab ID #D120-03) mg/L	**% RPD**
Nitrate	1.3	1.2	8.00
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.41J	0.39J	5.00
Chloride	500	550	9.52
Fluoride	0.95	0.95	<1
Sulfate	1800	1900	5.41

Anion	MW24042022 (Lab ID #D120-08) mg/L	MW24042022D (Lab ID #D120-10) mg/L	**% RPD**
Nitrate	0.048J	0.046J	4.26
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.55	0.54	1.83
Chloride	120	160	28.6
Fluoride	1.2	1.1	8.69
Sulfate	3.5	2.8	22.2

4.12.5. Raw data was submitted for all requested field samples. Sample TMW44042022 (Lab ID#22D120-05) and TMW46042022 (Lab ID#22D120-07) were designated to be reviewed as stage 3 data deliverable. Raw data for these samples together with all related QC samples was reviewed for stage 3 data validation. All 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 05-04-22 within 28-day holding time.

5.0 CONCLUSION

SDG #22D120 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 #22D131 Analytical Data Package

Publication Date: 08-08-22

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of twelve (12) water samples were collected on 04-13-22. EMAX Laboratories received the samples on 04-14-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. One sample from this sample delivery group, BGMW01042022(Lab ID#D131-06) 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 review. Two samples TMW63042022 (Lab ID#D131-08) and TMW27042022 (Lab ID#D131-11) were designated to be spiked as MS/MSD on the chain of custody. Results and recoveries of LCS/LCSD and MS/MSD was used for accuracy and precision. Raw data for method blank, LCS/LCSD and MS/MSD for each method were cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (12 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (9 samples)
EPA Method SW846 3535A/8330B: Nitroaromatics and Nitramines (10 samples)
EPA Method 8081B: Organochlorine Pesticides (9 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (5 sample)
EPA Method 8151A: Chlorinated herbicides (5 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (6 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (5 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 (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 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 # 22D131 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. Method SW3535A/8330B (Nitroaromatics and Nitramines) and method SW846 6850 (Perchlorate) was sub contracted by EMAX to GEL Laboratories in South Carolina.

A total of twelve (12) water samples were collected on 04-13-22. EMAX Laboratories received the samples on 04-14-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the data was subjected to validation to the equivalent of stage 3.

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD),

- Results and percent recoveries of Lab Control Samples (LCS/LCSD)
- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses and chromatograms for the sample/samples at Stage 3 deliverable and related QC samples.

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. One field sample BGMW01042022 (EMAX ID #D131-06) from this sample delivery group was designated for stage 3 data review. Raw data for this sample was evaluated comprehensively. Samples TMW63042022 (Lab ID#D131-08) and TMW27042022 (Lab ID#D131-11) were designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank, LCS/LCSD and MS/MSD was reviewed comprehensively. 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#22D131 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW30042022	22D131-01	04-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified Anions by IC
TMW50042022	22D131-02	04-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Modified TPH Gasoline; TPH as DRO Anions by IC
TMW50042022D	22D131-03	04-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Modified TPH Gasoline; TPH as DRO Anions by IC
TMW26042022	22D131-04	04-13-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22D131 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW39S042020	22D131-05	04-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified Anions by IC
BGMW01042022	22D131-06	04-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Organochlorine Pesticides Anions by IC
MW28042022	22D131-07	04-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Organochlorine Pesticides Anions by IC
TMW63042022	22D131-08	04-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Organochlorine Pesticides Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22D131 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
BGMW02042022	22D131-09	04-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Mercury Dissolved Mercury Dissolved Metals by ICP- MS Total Metals by ICP-MS Organochlorine Pesticides Anions by IC Perchlorate by 6850
MW25042022	22D131-10	04-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Organochlorine Pesticides Anions by IC
TMW27042022	22D131-11	04-13-22	S3VM	VOCs by SW5030B/8260C, Mercury Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
QC13042022TB3	22D131-12	04-13-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW63042022MS	22D131-08M	04-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Mercury Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Modified TPH Gasoline; TPH as DRO Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22D131 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW63042022MSD	22D131-08S	04-13-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Mercury Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Modified TPH Gasoline; TPH as DRO Anions by IC
TMW27042022MS	22D131-11M	04-13-22	S3VM	VOCs by SW5030B/8260C, Mercury Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW27042022MSD	22D131-11S	04-13-22	S3VM	VOCs by SW5030B/8260C, Mercury Dissolved 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
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW3535A/8330B	1.25-5µg/L
	Nitroglycerine & PETN	SW3535A/8330B	2.5µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in sixteen 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 sixteen 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 3535A/8330B: Explosives) =10/10X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =9/9X100=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) 6/6X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =5/5X100=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) =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 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 **3535A/8330B** for Nitrtoaromatics and Nitramine by LC/MS/MS
EPA Method **6850** for Perchlorate by HPLC/MS/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 fourteen samples. Water samples were collected on 04-13-22. Samples were analyzed on 04-21-22, 04-22-22, 04-26-22 and 04-27-22 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

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-05-22 and at the beginning of each analysis shift on 04-21-22, 04-22-22, 04-26-22 and 04-27-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-05-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-05-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.022), 2-Butanone (0.010), 2-Hexanone (0.062) and 4-Methyl 2-pentanone (0.086). 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 04-05-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-06-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standards were analyzed at the beginning and end of each analysis shift on 04-21-22, 04-22-22, 04-26-22 and 04-27-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-21-22 (I &II)	Continuing cal. Response factors 04-22-22 (I&II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-26-22 (I &II)	Continuing cal. Response factors 04-27-22 (I&II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-21-22) I&II	%Deviation from Initial calibration (04-22-22) I&II
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-26-22) I&II	%Deviation from Initial calibration (04-27-22) I&II
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for target analytes listed in the table below for each opening and closing daily standards. This should not affect the data quality.

Target analyte	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-21-22)		%Deviation from Initial calibration (04-22-22)	
		I	II	I	II
1,2-Dichloroethane	≤ 20%	--	22.3%*	--	32.5%*
2-Chloroethyvinylether	≤ 20%	20.8%*	30.6%*	--	--
Chloroform	≤ 20%	--	--	--	23.1%*
Bromodichloromethane	≤ 20%	--	--	--	22.3%*
Dibromomethane	≤ 20%	--	--	--	24.1%*
1,2,3-Trichlorobenzene	≤ 20%	--	--	--	21.6%*

*Failed maximum 20% D

Target analyte	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-26-22)		%Deviation from Initial calibration (04-27-22)	
		I	II	I	II
1,2-Dichlorometane	≤ 20%	21.3%*	20.8%*	--	--
2-Chloroethyvinylether	≤ 20%	25.0%*	34.7%*	38.9%*	37.5%*
Chloromethane	≤ 20%	--	--	--	23.7%*
2,2-Dichloropropane	≤ 20%	--	--	--	21.6%*

*Failed maximum 20% D

4.1.5. Quality Control samples reported consisted of four method blanks, four sets of LCS/LCSD and two sets of MS/MSD. Samples TMW63042022 and TMW27042022 were designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for each set of 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 for each set of LCS/LCSD and MS/MSD. However, 1,2-Dichloroethane recovery (129%) exceeded the required QC limit (73-128%) in LCS analyzed on 04-26-22. This analyte was not detected in any of the corresponding samples analyzed with this batch. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

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

4.1.6. **Field duplicate sample** and its associated sample: Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. No VOCs were detected in any of field sample or associated field duplicate sample except traces of Carbon disulfide in sample TMW50042022, as indicated in the table below:

Target analyte	TMW50042022 (Lab ID #D131-02) µg/L	TMW50042022D (Lab ID #D131-03) µg/L	**% RPD**
Carbon Disulfide	0.37J	U	200

4.1.7. Raw data was submitted for all samples. Sample BGMW01042022 (Lab ID# 22D131-06) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

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

4.2.1. Technical Holding Times

Holding time requirement was met for all nine samples. Water samples were collected on 04-13-22, extracted on 04-18-22 and were analyzed on 04-21-22 within required holding time.

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

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

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 04-21-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group. Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

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

√ denotes passing method acceptance limits

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

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

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

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

√ denotes passing method acceptance limits

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

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

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

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

4.2.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-21-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

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

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (04-21-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for 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 (04-21-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
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:

Target Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (04-21-22) I	%Deviation from Initial calibration (04-21-22) II
Nitrophenol	≤ 20	22.0%*	--
Benzoic acid	≤ 20	--	20.5%*

*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, one set of LCS/LCSD and MS/MSD. Sample TMW63042022 was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for all the QC samples reported were mostly within the project acceptance limits, except for two compounds in LCS as listed in the table below:

Target analyte	LCS	LCSD	QC Limit%
2,6-Dichlorophenol	49%*	55%	50-118
Phenol	47%*	50%	50-130

*Outside QC limit

Results and recoveries of LCSD was used to evaluate the QC for these compounds.

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

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

4.2.6. **Field duplicate sample** and its associated sample: Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. No SVOC target analytes was detected in any of field sample or associated field duplicate sample.

4.2.7. Raw data was submitted for all samples. Sample BGMW01042022 (Lab ID# 22D131-06) 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 nine water samples requested for this method. Water samples were collected on 04-13-22, extracted on 04-19-22 and analyzed on 04-20-22, 04-21-22 and 05-02-22 within required holding time.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 03-11-22, 03-12-22 and before sample analysis on 04-20-22, 04-21-22 and 05-02-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

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

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

4.3.3. Initial Calibration Verification and Continuing Calibration

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

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

A total of seven continuing calibration standards were analyzed at 10-injections interval on 04-20-22, 04-21-22 and 05-02-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. In all 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, one LCS and MS/MSD. Sample TMW63042022 was designated to be spiked as MS/MSD for this method. The Full pesticide target list was spiked and reported for LCS and MS/MSD. Percent recoveries (%R) were all within established QC limits. Results and recoveries of QC samples were confirmed with the reported raw data.

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

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. No organochlorine pesticide was detected in any of field sample or associated field duplicate sample.

4.3.6. Raw data was submitted for all samples. Sample BGMW01042022 (Lab ID# 22D131-06) 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.

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

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Five water samples were collected on 04-13-22, extracted on 04-19-22, and analyzed on 04-20-22.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-17-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-17-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-20-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for 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, one LCS and MS/MSD. Sample TMW63042022 was designated to be spiked as MS/MSD. Percent recoveries (%R) of LCS and MS/MSD were within the established QC limits.

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

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample: Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. No Polychlorinated Biphenyls (PCBs) was detected in any of field sample or associated field duplicate sample.

4.4.6. Raw data was submitted for all samples. Sample MW25042022(Lab ID#22D131-10) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Five water samples were collected on 04-13-22, extracted on 04-19-22 and analyzed on 04-21-22.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 04-19-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99776 was used for MCPP in column B.

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

4.5.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 04-19-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-21-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels except for Dinoseb (25%) in column 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 TMW63042022 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: Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. No Chlorinated Herbicides was detected in any of field sample or associated field duplicate sample.

4.5.6. Raw data was submitted for all samples. Sample MW25042022(Lab ID#22D131-10) 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 LC/MS/MS (EPA Method3535A/ 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 04-13-22, prepared on 04-20-22 and analyzed on 05-11-22 and 05-12-22 within holding time.

A High-Performance LC (HPLC) coupled with two Mass Detectors in tandem (LC/MS/MS) was used for analysis. Samples were sub-contracted to GEL Laboratories in South Carolina by EMAX Laboratory.

4.6.2. Initial Calibration

One initial calibration curve with seven concentration levels for each analyte was established on 05-10-22. Internal standard curve type was used for initial calibration and all the following analysis. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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) for all target analytes. Quadratic regression curve type was used for the following compound where %RSD exceeded the maximum 15 percent limit.

Target Analytes	**Quadratic Regression (Coeff. Of Determination) 05-10-22**
HMX	0.9992

4.6.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 05-11-22. Percent recoveries were within required method limits (70-130% of the true value).

Three continuing calibration standards were analyzed at the start and end of analysis shift on 05-11-22 and 05-12-22. Percent recoveries were all within required method limits (80-120% of the true value). Three Initial calibration blanks and continuing calibration blanks were all non-detect.

4.6.4. Quality Control samples consisted of one method blank, one LCS and MS/MSD. Sample TMW63042022 was designated to be spiked as MS/MSD. The entire explosive target lists were spiked and reported for LCS and MS/MSD. Percent recoveries (%R) were within the QAPP established QC limits for all target analytes in LCS. However, results of almost all analytes in MS/MSD were qualified due to low recoveries. Case narrative related this to parent sample probably being cross contaminated with the spiking standard. Therefore, result and recoveries of LCS was used for quality assurance. Raw data for blank 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, except sample MW28042022 (recovery reported as 39%, with acceptance limits being 44-142%).
3,4-Dinitrotoluene was used as surrogate.

4.6.5 Field duplicate sample and its associated sample**:** Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. No explosive was detected in the field sample or associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample BMW01042022 was designated as stage 3 data deliverable. Raw data foe this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.7.1. 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 04-13-22. Samples were analyzed on 04-18-22 and 04-19-22 within holding time requirement.

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

4.7.2. Initial Calibration

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

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

4.7.3. Initial Calibration Verification and Continuing Calibration

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

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-18-22 and 04-19-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.7.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63042022 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.

4.7.5. Field duplicate sample and its associated sample: Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. No TPH as Gasoline was detected in the field sample or associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample MW28042022(Lab ID#22D131-07) 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.

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

4.8.1. 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 04-13-22, extracted on 04-20-22 and analyzed on 04-21-22.

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

4.8.2. 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.

4.8.3. Initial 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%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-21-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group 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 TMW63042022 was designated for MS/MSD. Percent recoveries (%R) of LCS/LCSD and MS/MSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.8.5. Field duplicate sample and its associated sample: Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. Trace of TPH as DRO was detected in the field duplicate sample as indicated in the table below.

Target analyte	TMW50042022 (Lab ID #D131-02) mg/L	TMW50042022D (Lab ID #D131-03) mg/L	**% RPD**
TPH as DRO	U	0.26J	200

4.8.6. Raw data was submitted for all samples. Sample MW28042022(Lab ID#22D131-07) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Perchlorate by HPLC/MS/MS (EPA Method 6850 Modified)

4.9.1. 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 04-13-22. Samples were prepared on 05-02-22 and analyzed on 05-04-22.

A High-Performance LC coupled with two Mass Detectors (HPLC/MS/MS) was used for analysis. The sample for this method was sub-contracted by EMAX Laboratory to GEL (General Engineering Laboratories) in South Carolina.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 05-04-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.99985 (Perchlorate) and 0.99979 (perchlorate 101) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.05 – 10 µg/L.

Retention time for each isotope at each calibration level was within 0.2 minutes required by the method.

4.9.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 05-04-22. Percent recoveries were within required method limits (90-110% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of six daily standards were carried out on 05-04-22, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 90-110% limit.

4.9.4. Quality Control samples consisted of one method blank, one LCS and two sets of MS/MSD. Sample TMW63042022 and TMW27042022 were designated to be spiked as MS/MSD. Perchlorate was spiked and reported for LCS and MS/MSD. Percent recoveries (%R) were within the established QC limits LCS and MS/MSD (84-119%). Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Interference check sample (ICS) was analyzed and results were within acceptable limit (80-120% of the true value).

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.

4.9.5. Field duplicate sample and its associated sample: Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. Results for Perchlorate for field sample and sample duplicate are summarized in the table below:

Target analyte	TMW50042022 (Lab ID #D131-02) µg/L	TMW50042022D (Lab ID #D131-03) µg/L	**% RPD**
Perchlorate	2.20	2.28	3.57

4.10.6. Raw data was submitted for all samples. Sample BGMW01042022 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.

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

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eleven water samples requested for this method. Water samples were collected on 04-13-22. Samples were prepared (digested) for both total and dissolved metals on 04-18-22. Samples and QC samples were analyzed on 04-25-22 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.

4.10.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 04-25-22. 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. Five levels of concentration were used for initial calibration. 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 the 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.

4.10.3. Quality Control samples consisted of one method blank, one set of LCS/LCSD and two sets of MS/MSD for total and dissolved metals. Samples TMW63042022 and TMW27042022 were 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 summarized in the tables below. As a result, all the positive results will be qualified as estimated value “J” for these metals in the parent sample. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	TMW63042022 MS%	TMW63042022 MSD%	**QC Limit %**	TMW63042022 MS%	TMW63042022 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	√	√	87-115
Calcium	√	√	87-118	√	√	87-118
Chromium	√	√	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	45*	51*	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	700*	333*	85-117	467*	200*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

ANALYTE	Total Metals			Dissolved Metals		
	TMW27042022 MS%	TMW27042022 MSD%	**QC Limit %**	TMW27042022 MS%	TMW27042022 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	√	√	87-115
Calcium	103	73*	87-118	√	√	87-118
Chromium	√	√	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	√	√	85-118
Iron	√	√	87-118	√	√	87-118

ANALYTE	Total Metals			Dissolved Metals		
	TMW27042022 MS%	TMW27042022 MSD%	QC Limit %	TMW27042022 MS%	TMW27042022 MSD%	QC Limit%
Lead	√	√	88-115	√	√	88-115
Magnesium	√	√	83-118	√	√	83-118
Manganese	83*	67*	87-115	73*	127*	87-115
Nickel	√	√	85-117	√	√	85-117
Potassium	√	√	85-115	√	√	85-115
Selenium	√	√	80-120	√	√	80-120
Silver	√	√	85-116	√	√	85-116
Sodium	33*	200*	85-117	33*	-200*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	131*	131*	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. Samples TMW63042022 and TMW27042022 were 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 for both sets. 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.

4.10.4. Field duplicate sample and its associated sample: Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. Results and %RPD for field sample and associated field duplicate sample are listed in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW50042022 µg/L	TMW50042022D µg/L	% RPD	TMW50042022 µg/L	TMW50042022D µg/L	% RPD
Aluminum	3000	3100	3.28	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	3.1J	3.4J	9.23	1.9J	3.0J	44.9
Barium	59	61	3.33	16	16	<1
Beryllium	0.56J	0.58J	3.5	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	25000	27000	7.69	5300	5300	<1
Chromium	10	11	7.52	U	U	--
Cobalt	3.5J	3.7J	5.56	U	U	--
Copper	17	17	<1	U	5.3	200
Iron	2800	3000	6.9	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	TMW50042022 µg/L	TMW50042022D µg/L	% RPD	TMW50042022 µg/L	TMW50042022D µg/L	% RPD
Lead	2.2J	2.3J	4.44	U	U	--
Magnesium	1900	1900	<1	590	600	1.68
Manganese	750	810	7.69	14	13	7.41
Nickel	4.9J	5.6J	13.3	U	U	--
Potassium	810J	820J	1.23	630J	690J	9.1
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	520000	520000	<1	510000	510000	<1
Thallium	U	U	--	U	U	--
Vanadium	59	61	3.33	23	42	58.5
Zinc	27J	30J	6.9	U	U	--

4.10.5. Raw data was submitted for all samples. Sample BGMW01042022 (Lab ID#22D131-06) 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. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.11.1. 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 04-13-22. Samples were prepared (digested) on 04-25-22 (for Mercury) and 04-22-22 (for dissolved Mercury). Samples were analyzed on 04-25-22 for Mercury and on 04-22-22 for dissolved Mercury. All samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.11.2. Initial and continuing calibrations: The instrument calibrations for this method were acceptable. One blank and five standard levels were used for calibration curve at the beginning of each analysis day on 04-25-22 and 04-22-22. The correlation coefficient of at least 0.999112 and 0.999821 was calculated to show the linearity of each calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.11.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of one method blank, one set of LCS/LCSD and two sets of MS/MSD for Mercury and dissolved Mercury. Samples TMW63042022 and TMW27042022 were designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD and two sets of MS/MSD for both Mercury and dissolved Mercury. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.11.4. Field duplicate sample and its associated sample: Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. No Mercury was detected in the sample and associated field duplicate sample.

4.11.5. Raw data was submitted for all samples. Sample BGMW01042022 (Lab ID#22D131-06) 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.

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

4.12.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of ten water samples requested for this method. Water samples were collected on 04-13-22. Samples were analyzed on 04-14-22 and 04-15-22 for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride within the required 48-hour holding time. Samples were analyzed and re-analyzed at higher dilutions for Chloride and Sulfate on 04-26-22, 05-05-22 and 05-06-22 within 28-day holding times.

4.12.2. Initial and continuing calibration: Anions such as Chloride, Fluoride, Bromide, Nitrite, Nitrate, Orthophosphate and Sulfate were separated from water samples by Ion chromatography. The separated anions in their acid form (very conductive) were measured by conductivity. They were identified 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

03-04-22 and 05-04-22. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each anion. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 03-04-22 and 05-04-22. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 04-14-22, 04-15-22, 04-26-22, 05-05-22 and 05-06-22. A total of eighteen continuing calibration standards were analyzed with analysis and re-analysis of all samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.12.3. Quality Control Samples consisted of two method blanks, two sets of LCS/LCSD and MS/MSD. Sample TMW63042022 was designated to be spiked as MS/MSD. A third and fourth sets of QC samples (MB and LCS/LCD) was analyzed with dilutions and/or re-analysis of samples for Chloride and Sulfate. Recoveries of LCS/LCSD and MS/MSD were all within 90-110 % of spiked values for each anion. Percent RPDs were less than 20% for LCS/LCSD and MS/MSD.

4.12.4. Field duplicate sample and its associated sample: Field sample TMW50042022 was identified as field duplicate of sample TMW50042022D. Results for sample/sample duplicates are summarized in the table below:

Anion	TMW50042022 (Lab ID #D131-02) mg/L	TMW50042022D (Lab ID #D131-03) mg/L	**% RPD**
Nitrate	0.078J	0.070J	10.8
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	1.1	1.1	<1
Chloride	230	190	19.0
Fluoride	0.48	0.47	<1
Sulfate	620	570	9.52

4.12.5. Raw data was submitted for all requested field samples. Sample BGMW01042022 (Lab ID#22D131-06) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #22D131 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.

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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 #22D153 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 April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of eight (8) water samples were collected on 04-14-22. EMAX Laboratories received the samples on 04-15-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples was submitted for the requested analytical method. Sample TMW10042022
(Lab ID#D153-04) was designated to be reviewed as stage 3 deliverable. Raw data for this sample was compared to the reported summary tables and went through comprehensive data validation. No sample was designated to be spiked as MS/MSD. Recoveries and results of LCS/LCSD was used to evaluate accuracy and precision. Raw data for method blanks and LCS/LCSDs were also cross checked with the corresponding summary table results.

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

EPA Method 9056A: Anions by IC (8 samples)

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

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

The SDG # 22D153 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 eight (8) water samples were collected on 04-14-22. EMAX Laboratories received the samples on 04-15-22.

2.1 Data Reporting

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

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)
- Result and percent recoveries of MS/MSD, 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 TMW10042022 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. No sample was designated to be spiked as MS/MSD on the chain of custody. Raw data each set of LCS/LCSD was also reviewed. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);
- Technical holding times were confirmed for all samples with 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;
- LCS/LCSD results were evaluated; and
- Method blank results as well as surrogate recoveries were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#22D153 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
TMW31S042022	D153-01	04-14-22	S3VM	Anions by IC,
MW29042022	D153-02	04-14-22	S3VM	Anions by IC,
TMW08042022	D153-03	04-14-22	S3VM	Anions by IC,
TMW10042022	D153-04	04-14-22	S3VM	Anions by IC,
TMW51042022	D153-05	04-14-22	S3VM	Anions by IC,
QC14042022EB2	D153-06	04-14-22	S3VM	Anions by IC,
MW03042022	D153-07	04-14-22	S3VM	Anions by IC,
MW03042022D	D153-08	04-14-22	S3VM	Anions by IC,

TABLE 2-1

Summary of Analytical Parameters
USACE Wingate, New Mexico

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

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

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

TABLE 2-2

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

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

2.2.3 Laboratory and Field Blanks

The objective of laboratory and field blanks is to determine the presence and extent of contamination resulting from laboratory or field activities. Blanks reported here included method blank only. The result of analysis of method blank is discussed in Section 4.0 for this method. Samples were transported in one ice preserved cooler and was stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as 5.0˚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) =8/8X100=100%

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

4.0 DATA VALIDATION

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

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

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

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

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

The following Reason codes were applied in the report:

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

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

4.1.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of water samples requested for this method. A total of eight (8) water samples were collected on 04-14-22. Samples were analyzed on 04-15-22 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Some samples were re-analyzed on 05-05-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

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

Instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-04-22 and 05-04-22. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each anion. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 03-04-22 and 05-04-22. Recoveries were all within

90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 04-14-22 and 05-05-22. A total of seven continuing calibration standards were analyzed with analysis and re-analysis of all samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.12.3. Quality Control Samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. A second set of MB and LCS/LCSD was analyzed with Chloride and Sulfate analysis on 05-05-22. Recoveries of LCS/LCSDs were within 90-110 % of spiked values for each anion.

Percent RPDs were less than 20% for each set of LCS/LCSD for all anions.

4.12.4. Field duplicate sample and its associated sample: Sample MW03042022 was identified as field duplicate of MW03042022D. Results for sample/sample duplicate is summarized in the table below:

Anion	MW03042022 (Lab ID #D153-07) mg/L	MW03042022D (Lab ID #D153-08) mg/L	**% RPD**
Nitrate	6.2	6.2	<1
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.64	0.62	3.17
Chloride	370	300	20.9
Fluoride	0.48	0.47	2.11
Sulfate	1400	1300	7.41

4.12.5. Raw data was submitted for all requested field samples. Sample TMW10042022 (Lab ID#22D153-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #22D153 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 #22D165 Analytical Data Package

Publication Date: 08-05-22

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of fifteen (15) water samples were collected on 04-15-22. EMAX Laboratories received the samples on 04-16-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. One sample from this sample delivery group, TMW07042022(Lab ID#D165-02) 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 review. One sample TMW01042022 (Lab ID#D165-03) was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank, LCS/LCSD and MS/MSD for each method were cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (15 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (9 samples)
EPA Method SW846 3535A/8330B: Nitroaromatics and Nitramines (13 samples)
EPA Method 8081B: Organochlorine Pesticides (6 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (4 sample)
EPA Method 8151A: Chlorinated herbicides (4 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (7 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (6 samples)

EPA Method 6850: Perchlorate (12 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (14 samples)

EPA Method 7470A: Mercury & Dissolved Mercury (14 samples)
EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate (14 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 # 22D165 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. Method SW3535A/8330B (Nitroaromatics and Nitramines) and method SW846 6850 (Perchlorate) was sub contracted by EMAX to GEL Laboratories in South Carolina.

A total of fifteen (15) water samples were collected on 04-15-22. EMAX Laboratories received the samples on 04-16-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the data was subjected to validation to the equivalent of stage 3.

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD),

- Results and percent recoveries of Lab Control Samples (LCS/LCSD)
- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses and chromatograms for the sample/samples at Stage 3 deliverable and related QC samples.

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. One field sample TMW07042022 (EMAX ID #D165-02) from this sample delivery group was designated for stage 3 data review. Raw data for this sample was evaluated comprehensively. Sample TMW01042022 (Lab ID#D165-03) was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank, LCS/LCSD and MS/MSD was reviewed comprehensively. 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#22D165 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW23042022	22D165-01	04-15-22	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified Anions by IC
TMW07042022	22D165-02	04-15-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Anions by IC
TMW01042022	22D165-03	04-15-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified Anions by IC
TMW33042022	22D165-04	04-15-22	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
TMW22042022	22D165-05	04-15-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22D165 **Matrix: Water**

Field/Client ID	Lab ID	Date collected	Validation	Requested Methods of Analysis
TMW29042020	22D165-06	04-15-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified Anions by IC
TMW41042022	22D165-07	04-15-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Organochlorine Pesticides Anions by IC
TMW02042022	22D165-08	04-15-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW02042022D	22D165-09	04-15-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW55042022	22D165-10	04-15-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Organochlorine Pesticides Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22D165 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
BGMW11042022	22D165-11	04-15-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Organochlorine Pesticides Anions by IC
TMW04042022	22D165-12	04-15-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW62042022	22D165-13	04-15-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Organochlorine Pesticides Anions by IC
QC15042022EB3	22D165-14	04-15-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Mercury Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 Modified TPH Gasoline; TPH as DRO Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22D165 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
QC15042022TB5	22D165-15	04-15-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW01042022MS	22D165-03M	04-15-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Mercury Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW01042022MSD	22D165-03S	04-15-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Mercury Dissolved 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
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20μg/L, (Benzidine=40μg/L)
	Nitroaromatics &Nitramines	SW3535A/8330B	1.25-5μg/L
	Nitroglycerine & PETN	SW3535A/8330B	2.5μg/L
	Chlorinated Herbicides	SW8151A	1μg/L, (MCPA=40μg/L)
	Organochlorine Pesticides	SW8081B	0.1μg/L Methoxychlor =1.0μg/L Toxaphene =2.0μg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1μg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100μg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5μg/L,1μg/L,20μg/L,100μg/L,200μg/L
	Dissolved Mercury/Mercury	SW7470A	0.5μg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2μg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in fourteen 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 fourteen 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 5.2˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

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

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

Where:

RPD = Relative percent difference

R_1 = Result of the first duplicate or measured sample concentration

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

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

3.2.2 Accuracy

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

3.2.3 Completeness

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

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

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

Completeness (EPA Method 5030B/8260C: VOCs) =15/15X100=100%

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

Completeness (EPA Method 3535A/8330B: Explosives) =13/13X100=100%

Completeness (EPA Method 8081B: Organochlorine pesticides) =6/6X100=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) 7/7X100=100%

Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =6/6X100=100%

Completeness (EPA Method 6850: Perchlorate) =12/12X100=100%

Completeness (EPA Method 7470A: Mercury & Dissolved Mercury) =14/14X100=100%

Completeness (EPA Method 6020A: Dissolved and Total Metals) =14/14X100=100%

Completeness (EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate) =14/14X100=100%

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

4.0 DATA VALIDATION

This data review covers fifteen 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 **3535A/8330B** for Nitrtoaromatics and Nitramine by LC/MS/MS
EPA Method **6850** for Perchlorate by HPLC/MS/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 fifteen samples. Water samples were collected on 04-15-22. Samples were analyzed on 04-22-22, 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

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-21-22 and at the beginning of analysis shift on 04-22-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-21-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-21-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

Least square linear regression and/or quadratic regression curve type was used for the following compound where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Quadratic Regression (CCF) 04-21-22
Vinyl Chloride	0.9988

Target Analytes	Least Square Linear Regression (CCF) 04-21-22
Acetone	0.9984
Tetrahydrofuran	0.9972

Minimum average response factors for all the target compounds were within method's recommended values, except for: Acetone (0.049), and 2-Butanone (0.017). 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 04-21-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-22-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standards were analyzed at the beginning and end of analysis shift on 04-22-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-22-22 (I)	Continuing cal. Response factors 04-22-22 (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 (04-21-22) I	%Deviation from Initial calibration (04-22-22) 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 target analytes listed in the table below for opening and closing daily standards. This should not affect the data quality.

Target analyte	**%Deviation From Initial calibration (Acceptance Limit)**	**%Deviation from Initial calibration (04-22-22)**	
		I	**II**
Vinyl chloride	≤ 20%	25.1*	--
Trichlorofluoromethane	≤ 20%	--	34.6*

*Failed maximum 20% D

4.1.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD and one set of MS/MSD. Sample TMW01042022 was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the full compound list for LCS/LCSD and MS/MSD. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

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

4.1.6. **Field duplicate sample** and its associated sample: Field sample TMW02042022 was identified as field duplicate of sample TMW02042022D. No VOCs was detected in any of field sample or associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample TMW07042022 (Lab ID# 22D165-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

Traces of Bromodichloromethane, Chloroform, Bromoform 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 04-15-22, extracted on 04-20-22 and were analyzed on 04-25-22 within required holding time.

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

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

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 04-25-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

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

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

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

√ denotes passing method acceptance limits

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

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

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

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

√ denotes passing method acceptance limits

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

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

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

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

4.2.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-25-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

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

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	**Minimum response factor (Method limits)**	**Continuing cal. Response factors (04-25-22) I&II**
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for 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 (04-25-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target analyte list.

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

Target analyte	%LCS	%LCSD	**%RPD**	**%RPD Limit%**
4,6-Dinitro-2-methylphenol	88	110	21*	≤ 20

*Outside QC limit

Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision.

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 assigned to this method.

4.2.7. Raw data was submitted for all samples. Sample TMW07042022 (Lab ID# 22D165-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all six water samples requested for this method. Water samples were collected on 04-15-22, extracted on 04-21-22 and analyzed on 05-03-22, 05-04-22 and 05-11-22 within required holding time.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 03-11-22, 03-12-22 and before sample analysis on 05-03-22, 05-04-22 and 05-11-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

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

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

4.3.3. Initial Calibration Verification and Continuing Calibration

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

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

A total of eight continuing calibration standards were analyzed at 10-injections interval on 05-03-22, 05-04-22, 05-10-22 and 05-11-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A. Almost all target analytes in column B were qualified with high recovery (more than 20%). Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In all continuing calibration standards, one mid-point concentration of 20-40µg/L was injected.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked as 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. Results and recoveries of QC samples were confirmed with the reported raw data.

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

Surrogate recoveries were all within the method QC acceptance limits.

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

4.3.6. Raw data was submitted for all samples. Sample BGMW11042022(Lab ID#22D165-11) 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 04-15-22, extracted on 04-21-22, and analyzed on 04-25-22, within holding time requirement.

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 03-17-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-17-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-25-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

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

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) of LCS/LCSD 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 BGMW11042022(Lab ID#22D165-11) 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 04-15-22, extracted on 04-19-22 and analyzed on 04-21-22 within holding time.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 04-19-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99776 was used for MCPP in column B.

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

4.5.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 04-19-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-21-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels.

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

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

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

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

4.5.6. Raw data was submitted for all samples. Sample BGMW11042022(Lab ID#22D165-11) 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 LC/MS/MS (EPA Method3535A/ 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen (13) water samples requested for this method. Water samples were collected on 04-15-22, prepared on 04-22-22 and analyzed on 05-04-22, within holding time.

A High-Performance LC (HPLC) coupled with two Mass Detectors in tandem (LC/MS/MS) was used for analysis. Samples were sub-contracted to GEL Laboratories in South Carolina by EMAX Laboratory.

4.6.2. Initial Calibration

Two initial calibration curves with seven concentration levels for each analyte was established on 05-01-22 and 05-03-22. Internal standard curve type was used for initial calibration and all the following analysis. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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) for almost all target analytes.

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) 05-01-22**	**Least Square Linear Regression (CCF) 05-03-22**
RDX	0.9979	√
HMX	√	0.9992

4.6.3. Initial Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard (ICV) for all target analytes on 05-01-22 and 05-04-22. Percent recoveries were within required method limits (70-130% of the true value).

Three continuing calibration standards were analyzed at the start and end of analysis shift on 05-02-22 and 05-04-22. Percent recoveries were all within required method limits (80-120% of the true value). Three Initial calibration blanks and continuing calibration blanks were all non-detect.

4.6.4. Quality Control samples consisted of one method blank, one LCS and MS/MSD. Sample TMW01042022 was designated to be spiked as MS/MSD. The entire explosive target lists were spiked and reported for LCS and MS/MSD. Percent recoveries (%R) were within the QAPP established QC limits for all target analytes in LCS and MS/MSD. However, few analytes exceeded the QC limit requirement (percent recovery and/or %RPD) as summarized in the tables below:

Target analyte	TMW01042022MS	TMW01042022MSD	**QC Limit%**
PETN	59*%	63%	62-128
2,4-Dinitrotoluene	103%	109*%	80-108

*Exceeded QC limits

Target analyte	TMW01042022MS	TMW01042022MSD	%RPD	RPD Limit%
PETN	59*%	113%	63*	≤30
m-Nitrotoluene	66%	92%	32*	≤30
o-Nitrotoluene	66%	92%	49*	≤30
p-Nitrotoluene	56%	98%	54*	≤30

*Exceeded QC limits

Therefore, result and recoveries of LCS was used for quality assurance for these analytes. 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 (44-142%).

3,4-Dinitrotoluene was used as surrogate.

4.6.5 Field duplicate sample and its associated sample**:** Field sample TMW02042022 was identified as field duplicate of sample TMW02042022D. No explosives was detected in the field sample or associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample TMW07042022was designated as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.7.1. 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 04-15-22. Samples were analyzed on 04-19-22 within holding time requirement.

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

4.7.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-07-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.7.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-19-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

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 for this method. 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.

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

4.7.6. Raw data was submitted for all samples. Sample TMW07042022(Lab ID#22D165-02) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

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 04-15-22, extracted on 04-20-22 and analyzed on 04-21-22.

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

4.8.2. 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.

4.8.3. Initial 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%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-21-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group 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 as MS/MSD for this method. 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.

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

4.8.6. Raw data was submitted for all samples. Sample TMW07042022(Lab ID#22D165-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Perchlorate by HPLC/MS/MS (EPA Method 6850 Modified)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve samples requested for this method. Water samples were collected on 04-15-22. Samples were prepared on 05-03-22 and analyzed on 05-05-22, 05-06-22 and 05-09-22.

A High-Performance LC coupled with two Mass Detectors (HPLC/MS/MS) was used for analysis. The sample for this method was sub-contracted by EMAX Laboratory to GEL (General Engineering Laboratories) in South Carolina.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 05-04-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.99985 (Perchlorate) and 0.99979 (perchlorate 101) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.05 – 10 µg/L. A second initial calibration curve was run on 05-09-22 with some sample dilutions. All the QC requirements were satisfied.

Retention time for each isotope at each calibration level was within 0.2 minutes required by the method.

4.9.3. Initial Calibration Verification and Continuing Calibration

Initial calibrations were verified by a second source standard (ICV) on 05-04-22 and 05-09-22. Percent recoveries were within required method limits (90-110% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of twelve daily standards were carried out on 05-05-22, 05-06-22 and 05-09-22 bracketing the analyses and re-analysis of samples and all the QC samples. Recoveries of continuing calibration standards were within 90-110% limit. MDL verification standards were analyzed after each daily standard.

4.9.4. Quality Control samples consisted of one method blank, one LCS and one set of MS/MSD. Sample TMW01042022 was designated to be spiked as MS/MSD. Perchlorate was spiked and reported for LCS and MS/MSD. Percent recoveries (%R) were within the established QC limits LCS (84-119%). Recoveries of MS/MSD exceeded the upper QC limit as summarized in the table below:

Target analyte	TMW01042022MS	TMW01042022MSD	**QC Limit%**
Perchlorate	9250*%	19300*%	84-119

Therefore, the result for Perchlorate will be qualified as estimated value “J” in the parent sample. Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Interference check sample (ICS) was analyzed and results were within acceptable limit (80-120% of the true value).

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.

4.9.5. Field duplicate sample and its associated sample: Field sample TMW02042022 was identified as field duplicate of sample TMW02042022D. Results for Perchlorate for field sample and sample duplicate are summarized in the table below:

Target analyte	TMW02042022 µg/L	TMW02042022D µg/L	% RPD
Perchlorate	7.19	6.52	9.77

4.10.6. Raw data was submitted for all samples. Sample TMW07042022 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.

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

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for fourteen water samples requested for this method. Water samples were collected on 04-15-22. Samples were prepared (digested) for both total and dissolved metals on 04-19-22. Samples and QC samples were analyzed on 04-26-22 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.

4.10.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 04-26-22. 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. Five levels of concentration were used for initial calibration. 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 the 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.

4.10.3. Quality Control samples consisted of one method blank, one set of LCS/LCSD and one set of MS/MSD for total and dissolved metals. Sample TMW01042022 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 for the few metals as summarized in the table below. As a result, all the positive results will be qualified as estimated value "J" for these metals in the parent sample. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	TMW01042022 MS%	TMW01042022 MSD%	QC Limit %	TMW01042022 MS%	TMW01042022 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	√	√	87-115
Calcium	100	133*	87-118	33*	0*	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	103	120*	83-118	√	√	83-118
Manganese	√	√	87-115	√	√	87-115
Nickel	√	√	85-117	√	√	85-117
Potassium	√	√	85-115	√	√	85-115

ANALYTE	Total Metals			Dissolved Metals		
	TMW01042022 MS%	TMW01042022 MSD%	QC Limit %	TMW01042022 MS%	TMW01042022 MSD%	QC Limit%
Selenium	√	√	80-120	√	√	80-120
Silver	√	√	85-116	√	√	85-116
Sodium	67*	33*	85-117	-333*	-333*	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. Samples TMW01042022 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 for both sets of data. 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.

4.10.4. Field duplicate sample and its associated sample: Field sample TMW02042022 was identified as field duplicate of sample TMW02042022D. Results and %RPD for field sample and associated field duplicate sample are listed in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW02042022 (Lab ID#D165-08) µg/L	TMW02042022D (Lab ID#D165-09) µg/L	% RPD	TMW02042022 (Lab ID#D165-08) µg/L	TMW02042022D (Lab ID#D165-09) µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	7.5J	7.3J	2.70	7.9J	7.2J	9.27
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	24000	24000	<1	24000	26000	8.0
Chromium	U	U	--	1.2J	U	200
Cobalt	U	U	--	U	U	--
Copper	U	U	--	24	U	200
Iron	U	U	--	U	U	--
Lead	U	U	--	U	U	--
Magnesium	2800	2900	3.51	2900	2800	3.51
Manganese	U	U	--	U	U	--
Nickel	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	TMW02042022 (Lab ID#D165-08) µg/L	TMW02042022D (Lab ID#D165-09) µg/L	% RPD	TMW02042022 (Lab ID#D165-08) µg/L	TMW02042022D (Lab ID#D165-09) µg/L	% RPD
Potassium	1200J	1200J	<1	1300J	1300J	<1
Selenium	73	78	6.62	76	78	2.60
Silver	U	U	--	U	U	--
Sodium	1100000	1100000	<1	1100000	1100000	<1
Thallium	U	U	--	U	U	--
Vanadium	38	39	2.60	40	38	5.13
Zinc	U	U	--	U	U	--

4.10.5. Raw data was submitted for all samples. Sample TMW07042022 (Lab ID#22D165-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for fourteen (14) water samples requested for this method. Water samples were collected on 04-15-22. Samples were prepared (digested) and analyzed on 04-27-22 for Mercury and dissolved Mercury. All samples were preserved and filtered in the lab for dissolved Mercury analysis. Therefore, two sets of data were generated, one for Mercury and one for dissolved Mercury.

4.11.2. Initial and continuing calibrations: The instrument calibrations for this method were acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 04-27-22. The correlation coefficient of at least 0.999482 was calculated to show the linearity of each calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.11.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of one method blank, one set of LCS/LCSD and one set of MS/MSD for Mercury and dissolved Mercury. Samples TMW01042022 was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for both Mercury and dissolved Mercury. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.11.4. Field duplicate sample and its associated sample: Field sample TMW02042022 was identified as field duplicate of sample TMW02042022D. No Mercury was detected in the sample and associated field duplicate sample.

4.11.5. Raw data was submitted for all samples. Sample TMW07042022 (Lab ID#22D165-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.12.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of fourteen water samples requested for this method. Water samples were collected on 04-15-22. Samples were analyzed on 04-16-22 and 04-17-22 for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride within the required 48-hour holding time. Samples were analyzed and re-analyzed at higher dilutions for Chloride and Sulfate on 04-22-22, and 04-23-22 within 28-day holding times.

4.12.2. Initial and continuing calibration: Anions such as Chloride, Fluoride, Bromide, Nitrite, Nitrate, Orthophosphate and Sulfate were separated from water samples by Ion chromatography. The separated anions in their acid form (very conductive) were measured by conductivity. They were identified 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

03-04-22. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each anion. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 03-04-22. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 04-16-22, 04-17-22, 04-22-22 and 04-23-22. A total of fourteen continuing calibration standards were analyzed with analysis and re-analysis of all samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.12.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW01042022 was designated to be spiked as MS/MSD. A second and third set of QC (MB and LCS/LCD) was analyzed with dilutions and/or re-analysis of samples for Chloride and Sulfate. Recoveries of all three sets of LCS/LCSD were within 90-110 % of spiked values for each anion. Results and recoveries of MS/MSD were within the QC limits for Nitrate and Sulfate. All the remaining anions failed the required QC limits in MS/MSD as summarized in the table below:

Target analyte	TMW01042022MS	TMW01042022MSD	**QC Limit%**
Fluoride	114*%	115*%	82-112
Bromide	81*%	80*%	91-110
Nitrite	114*%	114*%	87-111
Chloride	96	83*%	87-111
Ortophosphate	80	79*%	80-116

*Outside QC limits

As a result, all the positive results will be qualified as estimated value “J” for these anions in the parent sample.

Percent RPDs were less than 20% for LCS/LCSD and MS/MSD.

4.12.4. Field duplicate sample and its associated sample: Field sample TMW02042022 was identified as field duplicate of sample TMW02042022D. Results for sample/sample duplicates are summarized in the table below:

Anion	TMW02042022 (Lab ID #D165-08) mg/L	TMW02042022D (Lab ID #D165-09) mg/L	**% RPD**
Nitrate	83	79	4.94
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	1.6	1.6	<1
Chloride	300	310	3.28
Fluoride	0.30	0.30	<1
Sulfate	1300	1300	<1

4.12.5. Raw data was submitted for all requested field samples. Sample TMW07042022 (Lab ID#22D165-02) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #22D165 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 #22D166 Analytical Data Package

Publication Date: 08-04-22

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of nine (9) water samples were collected on 04-14-22. EMAX Laboratories received the samples on 04-16-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. One sample from this sample delivery group, TMW10042022(Lab ID#D166-04) 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 review. No sample was designated to be spiked as MS/MSD on the chain of custody. 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 (4 samples)
EPA Method SW846 3535A/8330B: Nitroaromatics and Nitramines (7 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) (8 samples)
EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (7 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)

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 # 22D166 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. Method SW3535A/8330B (Nitroaromatics and Nitramines) and method SW846 6850 (Perchlorate) was sub contracted by EMAX to GEL Laboratories in South Carolina.

A total of nine (9) water samples were collected on 04-14-22. EMAX Laboratories received the samples on 04-16-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the data was subjected to validation to the equivalent of stage 3.

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if presented

- 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 TMW10042022 (EMAX ID #D166-04) 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. Therefore, results and recoveries of LCS/LCSD were used to evaluate accuracy and precision. Raw data for method blank, LCS/LCSD was reviewed comprehensively. 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#22D166 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW31S042022	22D166-01	04-14-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified
MW29042022	22D166-02	04-14-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
TMW08042022	22D166-03	04-14-22	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified TPH Gasoline; TPH as DRO
TMW10042022	22D166-04	04-14-22	S3VM	VOCs by SW5030B/8260C Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified TPH Gasoline; TPH as DRO
TMW51042022	22D166-05	04-14-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO

Site Name: Fort Wingate, New Mexico

SDG#22D166 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
QC14042022EB2	22D166-06	04-14-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
MW03042022	22D166-07	04-14-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO
MW03042022D	22D166-08	04-14-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO
QC14042022TB4	22D166-09	04-14-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

TABLE 2-1

Summary of Analytical Parameters
USACE Wingate, New Mexico

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW3535A/8330B	1.25-5µg/L
	Nitroglycerine & PETN	SW3535A/8330B	2.5µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements with qualifications if applied.

TABLE 2-2
Summary of Analytical Methods and Holding Time Requirements
USACE Wingate, New Mexico

ANALYSIS Method	**MATRIX**	**HOLDING TIME REQUIREMENT**	**DATA QUALIFIED AS "J"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	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 nine 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 5.2˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

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

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

Where:

RPD = Relative percent difference

R_1 = Result of the first duplicate or measured sample concentration

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

When analytes are present at concentrations below or near the quantitation limit, precision is measured, using MS/MSD, and/or LCS/LCSD results.

Precision results are discussed in Section 4.0 of this report.

3.2.2 Accuracy

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

3.2.3 Completeness

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

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

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

Completeness (EPA Method 5030B/8260C: VOCs) =9/9X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =4/4X100=100%
Completeness (EPA Method 3535A/8330B: Explosives) =7/7X100=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) 8/8X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =7/7X100=100%
Completeness (EPA Method 6850: Perchlorate) =8/8X100=100%
Completeness (EPA Method 7470A: Mercury & Dissolved Mercury) =8/8X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =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 **3535A/8330B** for Nitrtoaromatics and Nitramine by LC/MS/MS
EPA Method **6850** for Perchlorate by HPLC/MS/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury & 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. Nine water samples were collected on 04-14-22. Samples were analyzed on 04-22-22, 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

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-05-22 and at the beginning of analysis shift on 04-22-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-05-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-05-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

Minimum average response factors for all the target compounds were within method's recommended values, except for: Acetone (0.022), 2-Butanone (0.010), 4-Methyl-2-pentanone (0.086) and 2-Hexanone (0.062). 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 04-05-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-06-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standards were analyzed at the beginning and end of analysis shift on 04-22-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-22-22 (I)	Continuing cal. Response factors 04-22-22 (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 (04-22-22) I	%Deviation from Initial calibration (04-22-22) 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 target analytes listed in the table below for closing daily standards. This should not affect the data quality.

Target analyte	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-22-22)	
		I	II
1,2-Dichloroethane	≤ 20%	--	32.5%*
Tertt-Butylmethylether	≤ 20%	--	21.9%*
Chloroform	≤ 20%	--	23.1%*
Bromodichloromethane	≤ 20%	--	22.3%*
Dibromomethane	≤ 20%	--	24.1%*
1,2,3-Trichlorobenzene	≤ 20%	--	21.6%*

*Failed maximum 20% D

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

4.1.6. **Field duplicate sample** and its associated sample: Field sample MW03042022 was identified as field duplicate of sample MW03042022D. No VOCs were detected in any of field sample or associated field duplicate sample.
4.1.7. Raw data was submitted for all samples. Sample TMW10042022 (Lab ID# 22D166-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.
Traces of Bromodichloromethane, Chloroform, Bromoform 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 samples. Four water samples were collected on 04-14-22, extracted on 04-20-22 and were analyzed on 04-25-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.
Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 04-25-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

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

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

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

√ denotes passing method acceptance limits

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

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

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

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

√ denotes passing method acceptance limits

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

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

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

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

4.2.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-25-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

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

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (04-25-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for 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 (04-25-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√

Calibration Check Compounds (CCCs)	**% Deviation From Initial calibration (Acceptance Limit)**	**Accepted Deviation from Initial calibration (04-25-22) I& II**
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target analyte list.

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

Target analyte	%LCS	%LCSD	**%RPD**	**%RPD Limit%**
4,6-Dinitro-2-methylphenol	88	110	21*	≤ 20

*Outside QC limit

Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision.

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 assigned to this method.

4.2.7. Raw data was submitted for all samples. Sample MW29042022 (Lab ID# 22D166-02) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all five water samples requested for this method. Water samples were collected on 04-14-22, extracted on 04-19-22 and analyzed on 04-20-22, 04-21-22 and 05-02-22 within required holding time.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 03-11-22, 03-12-22 and before sample analysis on 04-20-22, 04-21-22 and 05-02-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

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

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

4.3.3. Initial Calibration Verification and Continuing Calibration

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

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

A total of seven continuing calibration standards were analyzed at 10-injections interval on 04-20-22, 04-21-22, and 05-02-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In all continuing calibration standards, one mid-point concentration of 20-40µg/L was injected.

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

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

Surrogate recoveries were all within the method QC acceptance limits.

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

4.3.6. Raw data was submitted for all samples. Sample MW29042022(Lab ID#22D166-02) was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. 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 04-14-22, extracted on 04-19-22, and analyzed on 04-20-22, within holding time requirement.

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 03-17-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-17-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-20-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for 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 LCS only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) of LCS was 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 MW29042022(Lab ID#22D166-02) with all related QC samples was selected to be 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 04-14-22, extracted on 04-19-22 and analyzed on 04-21-22.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 04-19-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99776 was used for MCPP in column B.

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

4.5.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 04-19-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-21-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels except for Dinoseb (25%) in column 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 and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. All herbicides target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits

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

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

4.5.6. Raw data was submitted for all samples. Sample MW29042022(Lab ID#22D166-02) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Nitroaromatics by LC/MS/MS (EPA Method3535A/ 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 04-14-22, prepared on 04-21-22 and analyzed on 05-09-22, within holding time.

A High-Performance LC (HPLC) coupled with two Mass Detectors in tandem (LC/MS/MS) was used for analysis. Samples were sub-contracted to GEL Laboratories in South Carolina by EMAX Laboratory.

4.6.2. Initial Calibration

Initial calibration curve with seven concentration levels for each analyte was established on 05-08-22. Internal standard curve type was used for initial calibration and all the following analysis. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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) for all target analytes.

4.6.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 05-08-22. Percent recoveries were within required method limits (70-130% of the true value).

Three continuing calibration standards were analyzed at the start and end of analysis shift on 05-09-22. Percent recoveries were all within required method limits (80-120% of the true value). However, few analytes exceeded the acceptance limits in the opening and closing continuing calibration standards as listed in the table below:

Target analyte	05-09-2022 Opening Daily St I	05-09-22 Mid Daily St II	05-09-2022 Closing Daily St III	**QC Limit%**
p-Nitrotoluene	128*%	√	128*%	80-120
o-Nitrotouene	√	√	128*%	80-120
Nitrobenzene	√	√	123*%	80-120

Since recoveries were greater than upper QC limit and no explosive was detected in any associated samples, this should not affect the quality of data.

Three Initial calibration blanks and continuing calibration blanks were all non-detect.

4.6.4. Quality Control samples consisted of one method blank and one LCS. No sample was designated to be spiked as MS/MSD. The lab reported a non-SDG sample as MS/MSD. This set of MS/MSD was not evaluated as part of this data. The entire explosive target lists were spiked and reported for LCS. Percent recoveries (%R) were within the QAPP established QC limits for all target analytes in LCS. Therefore, result and recoveries of LCS was used for quality assurance. 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 (44-142%).

3,4-Dinitrotoluene was used as surrogate.

4.6.5 Field duplicate sample and its associated sample**:** Field sample MW03042022 was identified as field duplicate of sample MW03042022D. No explosive was detected in the field sample or associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample TMW10042022was designated as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.7.1. 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 04-14-22. Samples were analyzed on 04-19-22 and 04-20-22 within holding time requirement.

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

4.7.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-07-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.7.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-19-22 and 04-20-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.7.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample MW29042022was spiked as MS/MSD for this method. 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.

4.7.5. Field duplicate sample and its associated sample: Field sample MW03042022 was identified as field duplicate of sample MW03042022D. No TPH as Gasoline was detected in the field sample or associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample TMW10042022(Lab ID#22D166-04) was designated as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

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 04-14-22, extracted on 04-20-22 and analyzed on 04-21-22 and 04-22-22 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.

4.8.2. 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.

4.8.3. Initial 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%

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-21-22 and 04-22-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked and reported as MS/MSD for this method. 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.

4.8.5. Field duplicate sample and its associated sample: Field sample MW03042022 was identified as field duplicate of sample MW03042022D. No TPH as DRO was detected in the field sample or associated field duplicate sample.

4.8.6. Raw data was submitted for all samples. Sample TMW10042022(Lab ID#22D166-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Perchlorate by HPLC/MS/MS (EPA Method 6850 Modified)

4.9.1. 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 04-14-22. Samples were prepared on 05-03-22 and analyzed on 05-05-22 and 05-09-22 within holding time.

A High-Performance LC coupled with two Mass Detectors (HPLC/MS/MS) was used for analysis. The sample for this method was sub-contracted by EMAX Laboratory to GEL (General Engineering Laboratories) in South Carolina.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 05-04-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.99985 (Perchlorate) and 0.99979 (perchlorate 101) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.05 – 10 µg/L. A second initial calibration curve was run on 05-09-22 with some sample re-analysis and/or dilutions. %RSD was less than 15% for Perchlorate. All the QC requirements were satisfied.

Retention time for each isotope at each calibration level was within 0.2 minutes required by the method.

4.9.3. Initial Calibration Verification and Continuing Calibration

Initial calibrations were verified by a second source standard (ICV) on 05-04-22 and 05-09-22. Percent recoveries were within required method limits (90-110% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of ten daily standards were carried out on 05-05-22 and 05-09-22 bracketing the analyses and re-analysis of samples and all the QC samples. Recoveries of continuing calibration standards were within 90-110% limit.

4.9.4. Quality Control samples consisted of one method blank and one LCS. A non-SDG sample was spiked and reported as MS/MSD. Therefore, the presented data was not evaluated. Perchlorate was spiked and reported for LCS. Percent recoveries (%R) were within the established QC limits LCS (84-119%).

Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Interference check sample (ICS) was analyzed and results were within acceptable limit (80-120% of the true value).

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.

4.9.5. Field duplicate sample and its associated sample: Field sample MW03042022 was identified as field duplicate of sample MW03042022D. No Perchlorate was detected in sample or field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample TMW10042022 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.

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

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 04-14-22. Samples were prepared (digested) for both total and dissolved metals on 04-20-22. Samples and QC samples were analyzed on 04-27-22 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.

4.10.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 04-27-22. 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. Five levels of concentration were used for initial calibration. 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 the 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.

4.10.3. Quality Control samples consisted of one method blank and one set of LCS/LCSD for total and dissolved metals. Sample TMW31S042022 was spiked and reported 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 for the few metals as summarized in the table below. As a result, all the positive results will be qualified as estimated value "J" for these metals in the parent sample.

ANALYTE	Total Metals			Dissolved Metals		
	TMW31S042022 MS%	TMW31S042022 MSD%	QC Limit %	NA	NA	QC Limit%
Aluminum	100	126*	84-117	NA	NA	84-117
Antimony	√	√	85-117	NA	NA	85-117
Arsenic	√	√	84-116	NA	NA	84-116
Barium	101	120*	86-114	NA	NA	86-114
Beryllium	√	√	83-121	NA	NA	83-121
Cadmium	√	√	87-115	NA	NA	87-115
Calcium	133*	167*	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	√	√	87-118	NA	NA	87-118
Lead	√	√	88-115	NA	NA	88-115
Magnesium	√	√	83-118	NA	NA	83-118
Manganese	√	√	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	267*	133*	85-117	NA	NA	85-117
Thallium	√	√	82-116	NA	NA	82-116
Vanadium	√	√	86-115	NA	NA	86-115
Zinc	119	121*	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 TMW31S042022 was used for serial dilution for total metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis for both sets of data. 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.

4.10.4. Field duplicate sample and its associated sample: Field sample MW03042022 was identified as field duplicate of sample MW03042022D. Results and %RPD for field sample and associated field duplicate sample are listed in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	MW03042022 µg/L	MW03042022D µg/L	% RPD	MW03042022 µg/L	MW03042022D µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	12	12	<1	12	11	8.69
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	76000	77000	1.31	80000	78000	2.53
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	20	U	200
Iron	160J	180J	11.7	U	U	--
Lead	U	U	--	U	U	--
Magnesium	14000	14000	<1	14000	14000	<1
Manganese	19	20	5.13	27	26	3.77
Nickel	U	U	--	U	U	--
Potassium	U	280J	200	310J	290JJ	6.66
Selenium	22	22	<1	21	21	<1
Silver	U	U	--	U	U	--
Sodium	990000	1000000	1.00	1000000	1000000	<1
Thallium	U	U	--	U	U	--
Vanadium	U	U	--	U	U	--
Zinc	U	U	--	U	U	--

4.10.5. Raw data was submitted for all samples. Sample TMW10042022 (Lab ID#22D166-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. 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 04-14-22. Samples were prepared (digested) on 04-25-22 and analyzed on 04-26-22 for Mercury. Samples were digested and analyzed on 04-29-22 for dissolved Mercury. All samples were preserved and filtered in the lab for dissolved Mercury analysis. Therefore, two sets of data were generated, one for Mercury and one for dissolved Mercury.

4.11.2. Initial and continuing calibrations: The instrument calibrations for this method were acceptable. One blank and five standard levels were used for calibration curve at the beginning of each analysis day on 04-26-22 and 04-29-22. The correlation coefficient of at least 0.999584 and 0.999557 was calculated to show the linearity of each calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.11.3. Quality Control: The data for all the QC samples were within acceptable control limits. 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 QC control limits for LCS/LCSD for both Mercury and dissolved Mercury. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.11.4. Field duplicate sample and its associated sample: Field sample MW03042022 was identified as field duplicate of sample MW03042022D. No Mercury was detected in the sample and associated field duplicate sample.

4.11.5. Raw data was submitted for all samples. Sample TMW10042022 (Lab ID#22D166-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #22D166 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of sixteen (16) water samples were collected on 04-18-22. EMAX Laboratories received the samples on 04-19-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples was submitted for the requested analytical method. Sample TMW52042022
(Lab ID#D177-01) was designated to be reviewed as stage 3 deliverable. Raw data for this sample was compared to the reported summary tables and went through comprehensive data validation. Sample TMW17042022 was designated to be spiked as MS/MSD. Recoveries and results of LCS/LCSD and MS/MSD was used to evaluate accuracy and precision. Raw data for method blanks MS/MSD and LCS/LCSDs were also cross checked with the corresponding summary table results.

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

EPA Method 9056A: Anions by IC (16 samples)

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

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

The SDG # 22D177 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 sixteen (16) water samples were collected on 04-18-22. EMAX Laboratories received the samples on 04-19-22.

2.1 Data Reporting

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

EMAX Laboratories provided the following information in one data package:

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

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

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for analytical method requested on the chain of custody were submitted for each sample. Sample TMW52042022 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 TMW17042022 was designated to be spiked as MS/MSD on the chain of custody. Raw data for MS/MSD with each set of LCS/LCSD was reviewed. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);
- Technical holding times were confirmed for all samples with 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;
- LCS/LCSD results were evaluated; and
- Method blank results as well as surrogate recoveries were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#22D177 **Matrix:**

Water

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
TMW52042022	D177-01	04-18-22	S3VM	Anions by IC,
MW33042022	D177-02	04-18-22	S3VM	Anions by IC,
TMW53042022	D177-03	04-18-22	S3VM	Anions by IC,
TMW14A042022	D177-04	04-18-22	S3VM	Anions by IC,
MW34042022	D177-05	04-18-22	S3VM	Anions by IC,
MW36S042022	D177-06	04-18-22	S3VM	Anions by IC,
MW26042022	D177-07	04-18-22	S3VM	Anions by IC,
MW36D042022	D177-08	04-18-2	S3VM	Anions by IC,
TMW17042022	D177-09	04-18-22	S3VM	Anions by IC,
MW36D042022D	D177-10	04-18-22	S3VM	Anions by IC,
TMW57042022	D177-11	04-18-22	S3VM	Anions by IC,
TMW61042022	D177-12	04-18-22	S3VM	Anions by IC,
TMW59042022	D177-13	04-18-22	S3VM	Anions by IC,
TMW28042022	D177-14	04-18-22	S3VM	Anions by IC,
FW31042022	D177-15	04-18-22	S3VM	Anions by IC,
QC18042022EB4	D177-16	04-18-22	S3VM	Anions by IC,
TMW17042022MS	D177-09M	04-18-22	S3VM	Anions by IC,
TMW17042022MSD	D177-09S	04-18-22	S3VM	Anions by IC,

TABLE 2-1

Summary of Analytical Parameters
USACE Wingate, New Mexico

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

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

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

TABLE 2-2

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

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

2.2.3 Laboratory and Field Blanks

The objective of laboratory and field blanks is to determine the presence and extent of contamination resulting from laboratory or field activities. Blanks reported here included method blank only. The result of analysis of method blank is discussed in Section 4.0 for this method. Samples were transported in one ice preserved cooler and was stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as 4.3˚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) =16/16X100=100%

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

4.0 DATA VALIDATION

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

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

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

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

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

The following Reason codes were applied in the report:

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

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

4.1.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of water samples requested for this method. A total of sixteen (16) water samples were collected on 04-18-22. Samples were analyzed on 04-19-22 and 04-20-22 within the required 48-hour holding time for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride. Samples were re-analyzed on 05-09-22 and 05-10-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

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

Instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-04-22 and 05-04-22. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each anion. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 03-04-22 and 05-04-22. Recoveries were all within

90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 04-19-22, 04-20-22, 05-09-22 and 05-10-22. A total of ten continuing calibration standards were analyzed with analysis and re-analysis of all samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected for the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.12.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD, MS/MSD and sample/sample duplicate. Sample TMW17042022 was designated to be spiked as MS/MSD. A second set of MB and LCS/LCSD was analyzed with Chloride and Sulfate analysis on 05-09-22. Recoveries of LCS/LCSDs were within 90-110 % of spiked values for each anion. Percent RPDs were less than 20% for each set of LCS/LCSD for all anions.

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

Anion	TMW17042022 MS%	TMW17042022 MSD%	% Acceptance limits
Chloride	101	112*	87-111
Bromide	82*	82*	91-110
Fluoride	116*	116*	88-112

*Outside Acceptance limits

4.12.4. Field duplicate sample and its associated sample: Sample MW36042022 was identified as field duplicate of MW36042022D. Results for sample/sample duplicate is summarized in the table below:

Anion	MW36D042022 (Lab ID #D177-08) mg/L	MW36D042022D (Lab ID #D177-10) mg/L	**% RPD**
Nitrate	0.074J	0.071J	4.14
Nitrite	U	U	--
Orthophosphate	0.62	0.68	9.23
Bromide	0.95	0.93	2.13
Chloride	110	110	<1
Fluoride	0.87	0.86	1.14
Sulfate	38	39	2.60

4.12.5. Raw data was submitted for all requested field samples. Sample TMW52042022 (Lab ID#22D177-01) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #22D177 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

6.0 REFERENCES

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

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

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

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

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

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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22D192 Analytical Data Package

Publication Date: 08-04-22

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of ten (10) water samples were collected on 04-19-22. EMAX Laboratories received the samples on 04-20-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. One sample from this sample delivery group, TMW64042022(Lab ID#D192-03) 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 review. No sample was designated to be spiked as MS/MSD on the chain of custody. Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision. Raw data for method blank and LCS/LCSD for each method were cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (10 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (6 samples)
EPA Method SW846 3535A/8330B: Nitroaromatics and Nitramines (7 samples)
EPA Method 8081B: Organochlorine Pesticides (6 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (2 sample)
EPA Method 8151A: Chlorinated herbicides (2 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (5 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (4 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)

EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate (9 samples)

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

All the requested samples were analyzed for each of the components listed in the corresponding EPA 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 # 22D192 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. Method SW3535A/8330B (Nitroaromatics and Nitramines) and method SW846 6850 (Perchlorate) was sub contracted by EMAX to GEL Laboratories in South Carolina.

A total of ten (10) water samples were collected on 04-19-22. EMAX Laboratories received the samples on 04-20-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the data was subjected to validation to the equivalent of stage 3.

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if presented

- 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 TMW64042022 (EMAX ID #D192-03) 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. Therefore, results and recoveries of LCS/LCSD were used to evaluate accuracy and precision. Raw data for method blank, LCS/LCSD was reviewed comprehensively. 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#22D192 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW24042022	22D192-01	04-19-22	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified Anions by IC
TMW45042022	22D192-02	04-19-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW64042022	22D192-03	04-19-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
TMW03042022	22D192-04	04-19-22	S3VM	VOCs by SW5030B/8260C Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Modified Anions by IC
TMW31D042022	22D192-05	04-19-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines 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#22D192 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW43042022	22D192-06	04-19-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW34042022	22D192-07	04-19-22	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Anions by IC
TMW34042022D	22D192-08	04-19-22	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Anions by IC
QC19042022EB5	22D192-09	04-19-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Anions by IC
QC19042022TB7	22D192-10	04-19-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

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

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 μg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20μg/L, (Benzidine=40μg/L)
	Nitroaromatics &Nitramines	SW3535A/8330B	1.25-5μg/L
	Nitroglycerine & PETN	SW3535A/8330B	2.5μg/L
	Chlorinated Herbicides	SW8151A	1μg/L, (MCPA=40μg/L)
	Organochlorine Pesticides	SW8081B	0.1μg/L Methoxychlor =1.0μg/L Toxaphene =2.0μg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1μg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100μg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5μg/L,1μg/L,20μg/L,100μg/L,200μg/L
	Dissolved Mercury/Mercury	SW7470A	0.5μg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2μg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in 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
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 eleven ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.7˚C and as high as 5.1˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

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

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

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) =10/10X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =6/6X100=100%
Completeness (EPA Method 3535A/8330B: Explosives) =7/7X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =6/6X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =2/2X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =2/2X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 5/5X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =4/4X100=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 (EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate) =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 **3535A/8330B** for Nitrtoaromatics and Nitramine by LC/MS/MS
EPA Method **6850** for Perchlorate by HPLC/MS/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 samples. Ten water samples were collected on 04-19-22. Samples were analyzed on 04-23-22, 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

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-05-22 and at the beginning of analysis shift on 04-23-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-05-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-05-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

Minimum average response factors for all the target compounds were within method's recommended values, except for: Acetone (0.022), 2-Butanone (0.010), 4-Methyl-2-pentanone (0.086) and 2-Hexanone (0.062). 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 04-05-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-06-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standards were analyzed at the beginning and end of analysis shift on 04-23-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-23-22 (I)	Continuing cal. Response factors 04-23-22 (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 (04-23-22) I	%Deviation from Initial calibration (04-23-22) 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 target analytes listed in the table below for closing daily standards. This should not affect the data quality.

Target analyte	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-23-22)	
		I	II
Hexachlorobutadiene	≤ 20%	22.5%*	--
2,2-Dichloropropane	≤ 20%	--	24.1%*
2-Chloroethyl vinyl ether	≤ 20%	--	23.6%*

*Failed maximum 20% D

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

compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the full compound list for LCS/LCSD. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.
Surrogate recoveries were all within the method's acceptable limits.

4.1.6. **Field duplicate sample** and its associated sample: Field sample TMW34042022 was identified as field duplicate of sample TMW34042022D. No VOCs was detected in any of field sample or associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample TMW64042022 (Lab ID# 22D192-03) 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 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 samples. Six water samples were collected on 04-19-22, extracted on 04-25-22 and were analyzed on 04-27-22 within required holding time.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.
Qualification notations, if any, will be summarized in result section; section 4.2.7.

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 04-27-22. It passed all the method assigned criteria. Tailing of

Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

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

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

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

√ denotes passing method acceptance limits

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

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

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

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

√ denotes passing method acceptance limits

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

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

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

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

4.2.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-27-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

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

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (04-27-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for 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 (04-27-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (04-27-22) I& II
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target analyte list except for the compound listed below in the opening continuing calibration standard:

Target Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (04-27-22) I	%Deviation from Initial calibration (04-27-22) I
2,4-Dinitrophenol	≤ 20	21.4%*	--

*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 only. No sample was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Quite a number of target analytes failed LCS and %RPD limits. Therefore, the data was evaluated by one LCSD only.

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 in samples listed in the table below:

Surrogate	Surrogate Recovery Limit%	TMW43042022 (D192-06)	QC19042022EB5 (D192-09)
2-Fluorobiphenyl	44-119	--	41%*
Nitrobenzene-D5	44-120	--	42%*
Phenol-D%	40-130	38%*	--

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

4.2.7. Raw data was submitted for all samples. Sample TMW64042022 (Lab ID# 22D192-03) 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. Six water samples were collected on 04-19-22, extracted on 04-22-22 and analyzed on 04-28-22 and 05-07-22 within required holding time.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 03-11-22, 03-12-22 and before sample analysis on 04-28-22, 04-29-22 and 05-07-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

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

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

4.3.3. Initial Calibration Verification and Continuing Calibration

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

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

A total of five continuing calibration standards were analyzed at 10-injections interval on 04-28-22, 04-29-22, and 05-07-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In all continuing calibration standards, one mid-point concentration of 20-40µg/L was injected.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked as 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. Results and recoveries of QC samples were confirmed with the reported raw data.

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

Surrogate recoveries were all within the method QC acceptance limits.

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

4.3.6. Raw data was submitted for all samples. Sample TMW64042022(Lab ID#22D192-03) 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. Two water samples were collected on 04-19-22, extracted on 04-22-22, and analyzed on 04-26-22, within holding time requirement.

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 03-17-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-17-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-26-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

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

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) of LCS/LCSD 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 TMW64042022(Lab ID#22D192-03) with all related QC samples was selected to be reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Two water samples were collected on 04-19-22, extracted on 04-22-22 and analyzed on 04-25-22.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 04-19-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99776 was used for MCPP in column B.

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

4.5.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 04-19-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-25-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels except for Picloram (35%) in column 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 and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. All herbicides target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits

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

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

4.5.6. Raw data was submitted for all samples. Sample TMW64042022(Lab ID#22D192-03) 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 LC/MS/MS (EPA Method3535A/ 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 04-19-22, prepared on 04-26-22 and analyzed on 05-04-22, within holding time.

A High-Performance LC (HPLC) coupled with two Mass Detectors in tandem (LC/MS/MS) was used for analysis. Samples were sub-contracted to GEL Laboratories in South Carolina by EMAX Laboratory.

4.6.2. Initial Calibration

One initial calibration curve with seven concentration levels for each analyte was established on 05-03-22. Internal standard curve type was used for initial calibration and all the following analysis. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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) for all target analytes, except one analyte (PETN). quadratic regression with correlation coefficient of 0.9988 was used for PETN.

4.6.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 05-03-22. Percent recoveries were within required method limits (70-130% of the true value).

Four continuing calibration standards were analyzed at the start and end of analysis shift on 05-04-22 and 05-05-22. Percent recoveries were all within required method limits (80-120% of the true value). However, two analytes exceeded the acceptance limits in the opening and closing continuing calibration standards run on 05-04-22, as listed in the table below:

Target analyte	05-04-2022		QC Limit%
	Daily St. I	Daily St. II	
p-Nitrotoluene	--	123%*	80-120
Nitroglycerine	122%*	--	80-120

Since recoveries were greater than upper QC limit and no explosive was detected in any associated samples, this should not affect the quality of data.

Three Initial calibration blanks and continuing calibration blanks were all non-detect.

4.6.4. Quality Control samples consisted of one method blank and one LCS. No sample was designated to be spiked as MS/MSD. The lab reported a non-SDG (non-client) sample as MS/MSD. This set of MS/MSD was not evaluated as part of this data. The entire explosive target lists were spiked and reported for LCS. Percent recoveries (%R) were within the QAPP established QC limits for all target analytes in LCS. Therefore, result and recoveries of LCS was used for quality assurance. 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 (44-142%).

3,4-Dinitrotoluene was used as surrogate.

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

4.6.6. Raw data was submitted for all samples. Sample TMW64042022 was selected as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.7.1. 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 04-19-22. Samples were analyzed on 04-20-22 within holding time requirement.

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

4.7.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-07-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.7.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-20-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.7.4. Quality Control samples consisted of one method blank one set of LCS/LCSD and MS/MSD. Sample TMW64042022 was spiked as MS/MSD for this method. 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.

4.7.5. Field duplicate sample and its associated sample: Field sample TMW34042022 was identified as field duplicate of sample TMW34042022D. No TPH as Gasoline was detected in the field sample or associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample TMW64042022(Lab ID#22D192-03) 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.

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

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 04-19-22, extracted on 04-22-22 and analyzed on 04-26-22 and 04-27-22 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.

4.8.2. 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.

4.8.3. Initial 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%

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-26-22 and 04-27-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was spiked as MS/MSD for this method. 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.

4.8.5. Field duplicate sample and its associated sample: Field sample TMW34042022 was identified as field duplicate of sample TMW34042022D. No TPH as DRO was detected in the field sample or associated field duplicate sample.

Trace of TPH as DRO was detected in the field duplicate sample as indicated in the table below.

Target analyte	TMW34042022 (Lab ID #D192-07) mg/L	TMW34042022D (Lab ID #D192-08) mg/L	**% RPD**
TPH as DRO	U	0.089J	200

4.8.6. Raw data was submitted for all samples. Sample TMW64042022(Lab ID#22D192-03) 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. Perchlorate by HPLC/MS/MS (EPA Method 6850 Modified)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for nine samples requested for this method. Water samples were collected on 04-19-22. Samples were prepared on 05-02-22 and analyzed on 05-04-22 and 05-05-22 within holding time.

A High-Performance LC coupled with two Mass Detectors (HPLC/MS/MS) was used for analysis. The sample for this method was sub-contracted by EMAX Laboratory to GEL (General Engineering Laboratories) in South Carolina.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 05-04-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.99985 (Perchlorate) and 0.99979 (perchlorate 101) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.05 – 10 µg/L. All the QC requirements were satisfied.

Retention time for each isotope at each calibration level was within 0.2 minutes required by the method.

4.9.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 05-04-22. Percent recoveries were within required method limits (90-110% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of seven daily standards were carried out on 05-04-22 and 05-05-22 bracketing the analyses and re-analysis of samples and all the QC samples. Recoveries of continuing calibration standards were within 90-110% limit.

4.9.4. Quality Control samples consisted of one method blank, one LCS and one set of MS/MSD. A non-SDG sample (client sample TMW63042022) was spiked and reported as MS/MSD. The sample was associated with SDG#22D131. Perchlorate was spiked and reported for LCS and MS/MSD. Percent recoveries (%R) were within the established QC limits

(84-119%). Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Interference check sample (ICS) was analyzed and results were within acceptable limit (80-120% of the true value).

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.

4.9.5. Field duplicate sample and its associated sample: Field sample TMW34042022 was identified as field duplicate of sample TMW34042022D. Results for Perchlorate are summarized in the table below for sample and sample duplicate:

Target analyte	TMW34042022 (Lab ID #D192-07) µg/L	TMW34042022D (Lab ID #D192-08) µg/L	**% RPD**
Perchlorate	0.308	0.314	1.93

4.10.6. Raw data was submitted for all samples. Sample TMW64042022 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. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.10.1. 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 04-19-22. Samples were prepared (digested) for both total and dissolved metals on 04-21-22. Samples and QC samples were analyzed on 04-28-22 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.

4.10.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 04-28-22. 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. Five levels of concentration were used for initial calibration. 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 the 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.

4.10.3. Quality Control samples consisted of one method blank and one set of LCS/LCSD for total and dissolved metals. Sample TMW24042022 was spiked and reported 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 for the two metals as summarized in the table below. As a result, all the positive results will be qualified as estimated value “J” for these metals in the parent sample.

ANALYTE	Total Metals			Dissolved Metals		
	TMW24042022 MS%	TMW24042022 MSD%	QC Limit %	NA	NA	QC Limit%
Aluminum	√	√	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	83*	87*	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	√	√	87-118	NA	NA	87-118
Lead	√	√	88-115	NA	NA	88-115
Magnesium	√	√	83-118	NA	NA	83-118

ANALYTE	Total Metals			Dissolved Metals		
	TMW24042022 MS%	TMW24042022 MSD%	QC Limit %	NA	NA	QC Limit%
Manganese	√	√	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	0.0*	0.0*	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. Samples TMW24042022 was used for serial dilution for total metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis for both sets of data. 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.

4.10.4. Field duplicate sample and its associated sample: Field sample TMW34042022 was identified as field duplicate of sample TMW34042022D. Results and %RPD for field sample and associated field duplicate sample are listed in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW34042022 µg/L	TMW34042022D µg/L	% RPD	TMW34042022 µg/L	TMW34042022D µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	13	13	<1	12	12	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	130000	130000	<1	130000	130000	<1
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	22	200
Iron	U	U	--	U	U	--
Lead	U	U	--	U	U	--
Magnesium	26000	27000	3.77	27000	27000	<1
Manganese	230	170	30.0	140	140	<1

ANALYTE	Total Metals			Dissolved Metals		
	TMW34042022 µg/L	TMW34042022D µg/L	% RPD	TMW34042022 µg/L	TMW34042022D µg/L	% RPD
Nickel	U	U	--	U	U	--
Potassium	600J	600J	<1	610J	650J	6.35
Selenium	120	120	<1	120	120	<1
Silver	U	U	--	U	U	--
Sodium	1300000	1300000	<1	1300000	1300000	<1
Thallium	U	U	--	U	U	--
Vanadium	U	U	--	U	U	--
Zinc	U	U	--	U	U	--

4.10.5. Raw data was submitted for all samples. Sample TMW64042022 (Lab ID#22D192-03) 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. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.11.1. 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 04-19-22. Samples were prepared (digested) on 04-25-22 and analyzed on 04-25-22 for Mercury. Samples were digested and analyzed on 04-29-22 for dissolved Mercury. All samples were preserved and filtered in the lab for dissolved Mercury analysis. Therefore, two sets of data were generated, one for Mercury and one for dissolved Mercury.

4.11.2. Initial and continuing calibrations: The instrument calibrations for this method were acceptable. One blank and five standard levels were used for calibration curve at the beginning of each analysis day on 04-25-22 and 04-29-22. The correlation coefficient of at least 0.999112 and 0.999557 was calculated to show the linearity of each calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.11.3. Quality Control: Data for all the QC samples were within acceptable control limits. 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 QC control limits for LCS/LCSD for both Mercury and dissolved Mercury. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.11.4. Field duplicate sample and its associated sample: Field sample TMW34042022 was identified as field duplicate of sample TMW34042022D. No Mercury was detected in the sample and associated field duplicate sample.

4.11.5. Raw data was submitted for all samples. Sample TMW64042022 (Lab ID#22D192-03) 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.

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

4.12.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of nine water samples requested for this method. Water samples were collected on 04-19-22. Samples were analyzed on 04-20-22 and 04-21-22 for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride within the required 48-hour holding time. Samples were re-analyzed at higher dilutions for Chloride and Sulfate on 04-22-22 within 28-day holding times.

4.12.2. Initial and continuing calibration: Anions such as Chloride, Fluoride, Bromide, Nitrite, Nitrate, Orthophosphate and Sulfate were separated from water samples by Ion chromatography. The separated anions in their acid form (very conductive) were measured by conductivity. They were identified 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 03-04-22. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout analysis. Percent RSD among calibration factors was less than 15%.

Calibration curve (concentration versus area count of each anion) was presented for each anion. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 03-04-22. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 04-20-22, 04-21-22 and 04-22-22. A total of ten continuing calibration standards were analyzed with analysis and re-analysis of all samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.12.3. Quality Control Samples consisted of two method blanks and two sets of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Recoveries of LCS/LCSDs were within 90-110 % of spiked values for each anion. Percent RPDs were less than 20% for each set of LCS/LCSD for all anions.

4.12.4. Field duplicate sample and its associated sample: Field sample TMW34042022 was identified as field duplicate of sample TMW34042022D. Results for sample/sample duplicates are summarized in the table below:

Anion	TMW34042022 (Lab ID #D192-07) mg/L	TMW34042022D (Lab ID #D192-08) mg/L	**% RPD**
Nitrate	68	66	2.98
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.98	1.0	2.02
Chloride	260	230	12.2
Fluoride	0.37	0.38	2.66
Sulfate	1800	1700	5.71

4.12.5. Raw data was submitted for all requested field samples. Sample TMW64042022 (Lab ID#22D192-03) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #22D192 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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6. Department of Defense Quality System Manual (DOD QSM), Version 5.3, 2019

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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #22D193 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 April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of seventeen (17) water samples were collected on 04-18-22. EMAX Laboratories received the samples on 04-20-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Two samples from this sample delivery group, TMW52042022(Lab ID#D193-01) and TMW61042022 (Lab ID#D193-12) were selected as stage 3 deliverable. Raw data for these samples was compared to the reported summary tables for each method and went through comprehensive data validation review. Sample TMW17042022 (Lab ID#D193-09) was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank, LCS/LCSD and MS/MSD for each method were cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (17 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (14 samples)
EPA Method SW846 3535A/8330B: Nitroaromatics and Nitramines (14 samples)
EPA Method 8081B: Organochlorine Pesticides (13 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (12 sample)
EPA Method 8151A: Chlorinated herbicides (12 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (13 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (12 samples)
EPA Method 6850: Perchlorate (13 samples)
EPA Method SW6020A: Dissolved and total Metals by ICP-MS (16 samples)
EPA Method 7470A: Mercury & Dissolved Mercury (16 samples)

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

All the requested samples were analyzed for each of the components listed in the corresponding EPA 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, one sample (TMW61042022) was extracted 25-days past 7-day required holding time for Explosives. The deviations, if any, are discussed in Section 4.0 for each method.

The SDG # 22D193 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. Method SW3535A/8330B (Nitroaromatics and Nitramines) and method SW846 6850 (Perchlorate) was sub contracted by EMAX to GEL Laboratories in South Carolina.

A total of seventeen (17) water samples were collected on 04-18-22. EMAX Laboratories received the samples on 04-20-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the data was subjected to validation to the equivalent of stage 3.

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if presented

- Results and percent recoveries of Lab Control Samples (LCS/LCSD)
- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses and chromatograms for the sample/samples at Stage 3 deliverable and related QC samples.

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. Two field samples, TMW52042022 (EMAX ID #D193-01) and TMW61042022 (EMAX ID #D193-12) from this sample delivery group was selected for stage 3 data review. Raw data for these samples was evaluated comprehensively. Sample TMW17042022
(Lab ID#D193-09) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for method blank, LCS/LCSD and MS/MSD was reviewed comprehensively. 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#22D193 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW52042022	22D193-01	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
MW33042022	22D193-02	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
TMW53042022	22D193-03	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
TMW14A042022	22D193-04	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS

Site Name: Fort Wingate, New Mexico

SDG#22D193 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
MW34042022	22D193-05	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
MW36S042022	22D193-06	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
MW26042022	22D193-07	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
MW36D042022	22D193-08	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO

Site Name: Fort Wingate, New Mexico

SDG#22D193 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW17042022	22D193-09	04-18-22	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
MW36D042022D	22D193-10	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
TMW57042022	22D193-11	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
TMW61042022	22D193-12	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO

Site Name: Fort Wingate, New Mexico

SDG#22D193 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW59042022	22D193-13	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
TMW28042022	22D193-14	04-18-22	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS
FW31042022	22D193-15	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS
QC18042022EB4	22D193-16	04-18-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
QC18042022TB6	22D193-17	04-18-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW17042022MS	22D193-09M	04-18-22	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
TMW17042022MSD	22D193-09S	04-18-22	S3VM	VOCs by SW5030B/8260C, 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	SW3535A/8330B	1.25-5μg/L
	Nitroglycerine & PETN	SW3535A/8330B	2.5μg/L
	Chlorinated Herbicides	SW8151A	1μg/L, (MCPA=40μg/L)
	Organochlorine Pesticides	SW8081B	0.1μg/L Methoxychlor =1.0μg/L Toxaphene =2.0μg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1μg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100μg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5μg/L,1μg/L,20μg/L,100μg/L,200μg/L
	Dissolved Mercury/Mercury	SW7470A	0.5μg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2μg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements with qualifications if applied.

TABLE 2-2

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

ANALYSIS Method	MATRIX	HOLDING TIME REQUIREMENT	DATA QUALIFIED AS "J"
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	Holding times were met for all **samples except one***
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

***Sample TMW61042022** was extracted for explosives on 05-19-22; 25 days past holding time

2.2.3 Laboratory and Field Blanks

The objective of laboratory and field blanks is to determine the presence and extent of contamination resulting from laboratory or field activities. Blanks reported here included method and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in twenty ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 0.5˚C and as high as 5.7˚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:

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

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) =17/17X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =14/14X100=100%
Completeness (EPA Method 3535A/8330B: Explosives) =14/14X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =13/13X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =12/12X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =12/12X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 13/13X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =12/12X100=100%
Completeness (EPA Method 6850: Perchlorate) =13/13X100=100%
Completeness (EPA Method 7470A: Mercury & Dissolved Mercury) =16/16X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =16/16X100=100%

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

4.0 DATA VALIDATION

This data review covers 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 **3535A/8330B** for Nitrtoaromatics and Nitramine by LC/MS/MS
EPA Method **6850** for Perchlorate by HPLC/MS/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury & 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. Seventeen water samples were collected on 04-18-22. Samples were analyzed on 04-23-22, 04-26-22 and 04-27-22 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

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-05-22 and at the beginning of each analysis shift on 04-23-22, 04-26-22 and 04-27-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-05-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-05-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

Minimum average response factors for all the target compounds were within method's recommended values, except for: Acetone (0.022), 2-Butanone (0.010), 4-Methyl-2-pentanone (0.086) and 2-Hexanone (0.062). 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 04-05-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-06-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standards were analyzed at the beginning and end of each analysis shift on 04-23-22, 04-26-22 and 04-27-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-23-22 (I&II)	Continuing cal. Response factors 04-26-22 (I&II)	Continuing cal. Response factors 04-27-22 (I&II)
Chloromethane	≥ 0.10	√	√	√
1,1-Dichloroethane	≥ 0.20	√	√	√
Chlorobenzene	≥ 0.50	√	√	√
Bromoform	≥ 0.10	√	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√	√

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-23-22) I&II	%Deviation from Initial calibration (04-26-22) I&II	%Deviation from Initial calibration (04-27-22) I&II
Vinyl chloride	≤ 20%	√	√	√
1,1-Dichloroethene	≤ 20%	√	√	√
Chloroform	≤ 20%	√	√	√
1,2-Dichloropropane	≤ 20%	√	√	√
Toluene	≤ 20%	√	√	√
Ethyl benzene	≤ 20%	√	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for target analytes listed in the table below for each opening and closing daily standards. This should not affect the data quality.

Target analyte	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-23-22)		%Deviation from Initial calibration (04-26-22)		%Deviation from Initial calibration (04-27-22)	
		I	II	I	II	I	II
Hexachlorobutadiene	≤ 20%	22.5*	√	√	√	√	√
2,2-Dichloropropane	≤ 20%	√	24.1*	√	21.1*	√	21.6*
2-Chloroethyl vinyl ether	≤ 20%	√	23.6*	25.0*	34.7*	38.9*	37.5*
1,2-Dichloroethane	≤ 20%	√	√	21.3*	20.8*	√	√
2-Chloromethane	≤ 20%	√	√	√	√	√	23.7*

*Failed maximum 20% D

4.1.5. Quality Control samples reported consisted of three method blanks, three sets of LCS/LCSD and MS/MSD. Sample TMW17042022 was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the full compound list for LCS/LCSD and MS/MSD. 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 1,2-Dichloroethane-D4 in the two samples as shown in the table below:

Surrogate	Surrogate Recovery Limit%	TMW61042022 (D193-12)	TMW28042022 (D193-14)
1,2-Dichloroethane-D4	81-118	121%*	119%*

4.1.6. **Field duplicate sample** and its associated sample: Field sample MW36D042022 was identified as field duplicate of sample MW36D042022D. No VOCs was detected in each of field sample or associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Samples TMW52042022 (Lab ID# 22D193-01) and TMW61042022 (Lab ID# 22D193-12) were selected 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 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 samples. Fourteen water samples were collected on 04-18-22, extracted on 04-25-22 and were analyzed on 04-27-22 within required holding time.

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

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

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 04-27-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

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

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

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

√ denotes passing method acceptance limits

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

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

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

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

√ denotes passing method acceptance limits

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

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

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

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

4.2.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-27-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

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

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (04-27-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for 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 (04-27-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
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 analyte list except for the compound listed below in the opening continuing calibration standard:

Target Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (04-27-22) I	%Deviation from Initial calibration (04-27-22) I
2,4-Dinitrophenol	≤ 20	21.4%*	--

*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 only. No sample was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Quite a number of target analytes failed LCS and %RPD limits. Therefore, the data was evaluated by one LCSD only.

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 in samples listed in the table below:

Surrogate	Surrogate Recovery Limit %	TMW52042022 (D193-01)	MW33042022 (D193-02)	TMW53042022 (D193-03)	TMW14A042022 (D193-04)	FW31042022 (D193-15)
2-Fluorobiphenyl	44-119	35%*	--	40%*	36%	35%*
2-Fluorophenol	19-119	9%*	16%*	--	--	--
Nitrobenzene-D5	44-120	37%*	--	42%*	39%*	35%*
Phenol-D%	40-130	20%*	32%*	38%*	35%*	36%*

*Failed surrogate acceptance limits

4.2.6. **Field duplicate sample** and its associated sample: Field sample MW36D042022 was identified as field duplicate of sample MW36D042022D. No SVOCs was detected in each of field sample or associated field duplicate sample.

4.2.7. Raw data was submitted for all samples. Samples TMW52042022 (Lab ID# 22D193-01) and TMW61042022 (Lab ID# 22D193-12) were selected to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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. Thirteen water samples were collected on 04-18-22, extracted on 04-22-22 and analyzed on 04-28-22, 04-29-22 and 05-05-22 within required holding time.

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

DDT- Endrin breakdown mix was analyzed prior to each initial calibration on 03-11-22, 03-12-22, 04-28-22 and before sample analysis on 04-28-22, 04-29-22 and 05-05-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Two sets of initial calibration curves were analyzed with eight levels of concentration for each pesticide on 03-11-22, 03-12-22 and 04-28-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 03-12-22 and 04-29-22 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene.

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

4.3.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 03-12-22, 03-15-22 and 05-02-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

A total of seven continuing calibration standards were analyzed at 10-injections interval on 04-28-22, 04-29-22, and 05-05-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A. One analyte, (alpha-BHC) exceeded 20% limit in two standards run on 04-29-22 in column B. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In all continuing calibration standards, one mid-point concentration of 20-40µg/L was injected.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked as 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. Results and recoveries of QC samples were confirmed with the reported raw data.

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

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Field sample MW36D042022 was identified as field duplicate of sample MW36D042022D. No pesticides was detected in each of field sample or associated field duplicate sample.

4.3.6. Raw data was submitted for all samples. Samples TMW52042022 (Lab ID# 22D193-01) and TMW61042022 (Lab ID# 22D193-12) were selected to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Twelve water samples were collected on 04-18-22, extracted on 04-22-22, and analyzed on 04-26-22, within holding time requirement.

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 03-17-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-17-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-26-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

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

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

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

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample and its associated sample: Field sample MW36D042022 was identified as field duplicate of sample MW36D042022D. No Polychlorinated Biphenyls (PCBs) was detected in each of field sample or associated field duplicate sample.

4.4.6. Raw data was submitted for all samples. Samples TMW52042022 (Lab ID# 22D193-01) and TMW61042022 (Lab ID# 22D193-12) were selected to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Twelve water samples were collected on 04-18-22, extracted on 04-22-22 and analyzed on 04-25-22, 04-29-22 and 04-30-22.

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

4.5.2. Initial Calibration

Two initial calibrations were performed with eight levels of concentration for each herbicide on 04-19-22 and 04-28-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99776 and 0.99922 was used for MCPP in column B.

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

4.5.3. Initial Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard (ICV) for all target herbicides on 04-19-22 and 04-28-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-25-22, 04-29-22 and 04-30-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels except for Picloram (35%) in column B in the closing daily standard run on 04-25-22.

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

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

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

4.5.5. Field duplicate sample and its associated sample: Field sample MW36D042022 was identified as field duplicate of sample MW36D042022D. No Chlorinated Herbicides was detected in each of field sample or associated field duplicate sample.

4.5.6. Raw data was submitted for all samples. Samples TMW52042022 (Lab ID# 22D193-01) and TMW61042022 (Lab ID# 22D193-12) were selected to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.6. Nitroaromatics by LC/MS/MS (EPA Method3535A/ 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen (13) out of fourteen (14) water samples requested for this method. Water samples were collected on 04-18-22, prepared on 04-25-22 and analyzed on 05-11-22 and 05-12-22, within holding time. However, one sample, TMW61042022 (EMAX ID# D193-12) was extracted on 05-19-22, twenty-five days past holding time. As shown in the communications and mentioned in the case narrative, this sample was put on hold in the GEL Laboratory and was extracted on 05-19-22 after the labeling issue was resolved.

A High-Performance LC (HPLC) coupled with two Mass Detectors in tandem (LC/MS/MS) was used for analysis. Samples were sub-contracted to GEL Laboratories in South Carolina by EMAX Laboratory.

4.6.2. Initial Calibration

One initial calibration curve with seven concentration levels for each analyte was established on 05-10-22. Internal standard curve type was used for initial calibration and all the following analysis. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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) for all target analytes, except one analyte (HMX). quadratic regression curve fit with correlation coefficient of 0.9992 was used for HMX. A second initial calibration was run on 05-19-22, corresponding to the sample extracted out of holding time. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent) for all target analytes, except one Nitroglycerin. Quadratic regression curve fit with correlation coefficient of 0.9989 was used for this compound.

4.6.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 05-11-22 and 05-19-22. Percent recoveries were within required method limits (70-130% of the true value).

Five continuing calibration standards were analyzed at the start and end of analysis shift on 05-11-22 and 05-12-22. Percent recoveries were all within required method limits (80-120% of the true value) in the three standards associated with the original runs. However, few analytes exceeded the acceptance limits in the last two standards analyzed on 05-12-22 as listed in the table below:

Target analyte	05-12-2022 Daily St I	05-12-2022 Daily St II	**QC Limit%**
o-Nitrotoluene	72.9%*	70%*	80-120
p-Nitrotoluene	75.5%*	73%*	80-120
m-Nitrotoluene	68%*	72.1%*	80-120
PETN	74%*	78.5%*	80-120
Nitroglycerine	--	143%*	80-120

Associated sample (TMW59042022), was re-analyzed on 05-12-22 and the recoveries of the target of interest (RDX) was within QC limit in both standards. Therefore, it does not affect the data quality. Two more continuing calibration standards were analyzed with one sample (TMW61042022), with missed holding time. Recoveries were mostly within 80-120% except for PETN (76.6%) and HMX (74.0%) in the opening standard run on 05-24-22.

Initial calibration blanks and continuing calibration blanks were all non-detect.

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 entire explosive target lists were spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the QAPP established QC limits for all target analytes. 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 (44-142%).

3,4-Dinitrotoluene was used as surrogate.

4.6.5 Field duplicate sample and its associated sample**:** Field sample MW36D042022 was identified as field duplicate of sample MW36D042022D. No explosives were detected in each of field sample or associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample TMW52042022 was selected 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. As mentioned in section 4.6.1, sample TMW61042022 was extracted twenty-five days past required holding time, therefore the data presented for this sample was qualified as "UJ" for any positive result and "UX" for all non-detects. GEL (laboratory in charge of the analysis for this method), reported all results as "UH" for this sample, (exceeded holding time).

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

4.7.1. 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 04-18-22. Samples were analyzed on 04-20-22 and 04-21-22 within holding time requirement.

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

4.7.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-07-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.7.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-20-22 and 04-21-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.7.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was spiked as MS/MSD for this method. 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.

4.7.5. Field duplicate sample and its associated sample: Field sample MW36D042022 was identified as field duplicate of sample MW36D042022D. No TPH as Gasoline was detected in the field sample or associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Samples TMW52042022(Lab ID#22D193-01) and TMW61042022 (Lab ID#22D193-12) was selected to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.8.1. 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 04-18-22, extracted on 04-22-22 and analyzed on 04-25-22, 04-26-22 and 04-27-22 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.

4.8.2. 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.

4.8.3. Initial 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%

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-25-22, 04-26-22 and 04-27-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD for this method. 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.

4.8.5. Field duplicate sample and its associated sample: Field sample MW36D042022 was identified as field duplicate of sample MW36D042022D. No TPH as DRO was detected in the field sample or associated field duplicate sample.

4.8.6. Raw data was submitted for all samples. Samples TMW52042022(Lab ID#22D193-01) and TMW61042022 (Lab ID#22D193-12) was selected to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Perchlorate by HPLC/MS/MS (EPA Method 6850 Modified)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for thirteen samples requested for this method. Water samples were collected on 04-18-22. Samples were prepared on 05-03-22 and analyzed on 05-06-22 within holding time.

A High-Performance LC coupled with two Mass Detectors (HPLC/MS/MS) was used for analysis. The sample for this method was sub-contracted by EMAX Laboratory to GEL (General Engineering Laboratories) in South Carolina.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 05-04-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.99985 (Perchlorate) and 0.99979 (perchlorate 101) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.05 – 10 µg/L. All the QC requirements were satisfied.

Retention time for each isotope at each calibration level was within 0.2 minutes required by the method.

4.9.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 05-04-22. Percent recoveries were within required method limits (90-110% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of three daily standards were carried out on 05-06-22 bracketing the analyses and re-analysis of samples and all the QC samples. Recoveries of continuing calibration standards were within 90-110% limit.

4.9.4. Quality Control samples consisted of one method blank, one LCS and one set of MS/MSD. Sample TMW17042022 was spiked and reported as MS/MSD. Perchlorate was spiked and reported for LCS. Percent recoveries (%R) were within the established QC limits for LCS and MS/MSD (84-119%). Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Interference check sample (ICS) was analyzed and results were within acceptable limit (80-120% of the true value).

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.

4.9.5. Field duplicate sample and its associated sample: Field sample MW36D042022 was identified as field duplicate of sample MW36D042022D. No Perchlorate was detected in each of sample or field duplicate sample.

4.10.6. Raw data was submitted for all samples. Samples TMW52042022 and TMW61042022 were selected to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.10.1. 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 04-18-22. Samples were prepared (digested) for both total and dissolved metals on 04-21-22. Samples and QC samples were analyzed on 05-02-22 and 05-03-22for 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.

4.10.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of each analysis day on 05-02-22 and 05-03-22. 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. Five levels of concentration were used for each initial calibration. 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 the 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.

4.10.3. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW17042022 was spiked and reported as MS/MSD for total and dissolved metals. 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 two metals as summarized in the table below. As a result, all the positive results will be qualified as estimated value "J" for these metals in the parent sample.

ANALYTE	Total Metals			Dissolved Metals		
	TMW17042022 MS%	TMW17042022 MSD%	QC Limit %	TMW17042022 MS%	TMW17042022 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		
	TMW17042022 MS%	TMW17042022 MSD%	**QC Limit %**	TMW17042022 MS%	TMW17042022 MSD%	**QC Limit%**
Cadmium	√	√	87-115	√	√	87-115
Calcium	√	√	87-118	77*	100	87-118
Chromium	√	√	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	101	126*	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	800*	-100*	85-117	767*	567*	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. Samples TMW17042022 was used for serial dilution for total and dissolved metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis for both sets of data. 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.

4.10.4. Field duplicate sample and its associated sample: Field sample MW36D042022 was identified as field duplicate of sample MW36D042022D. Results and %RPD for field sample and associated field duplicate sample are listed in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	MW36D042022 µg/L	MW36D042022D µg/L	**% RPD**	MW36D042022 µg/L	MW36D042022D µg/L	**% RPD**
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	190	190	<1	160	160	<1
Beryllium	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	MW36D042022 µg/L	MW36D042022D µg/L	% RPD	MW36D042022 µg/L	MW36D042022D µg/L	% RPD
Cadmium	U	U	--	U	U	--
Calcium	37000	35000	5.56	38000	36000	5.41
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	870J	840J	3.51	U	U	--
Lead	U	U	--	U	U	--
Magnesium	13000	13000	<1	13000	13000	<1
Manganese	220	220	<1	200	200	<1
Nickel	U	U	--	U	U	--
Potassium	830J	790J	<1	840J	820J	2.41
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	580000	560000	3.51	600000	570000	5.13
Thallium	U	U	--	U	U	--
Vanadium	U	U	--	2.1J	U	200
Zinc	U	U	--	U	U	--

4.10.5. Raw data was submitted for all samples. Samples TMW52042022 (Lab ID# D193-01) and TMW61042022 (Lab ID# D193-12) were selected to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. 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 04-18-22. Samples were prepared (digested) on 05-02-22 and analyzed on 05-02-22 for Mercury and dissolved Mercury. All samples were preserved and filtered in the lab for dissolved Mercury analysis. Therefore, two sets of data were generated, one for Mercury and one for dissolved Mercury.

4.11.2. Initial and continuing calibrations: The instrument calibrations for this method were acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 05-02-22. The correlation coefficient of at least 0.999779 was calculated to show the linearity of each calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.11.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of one method blank one set of LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Sample TMW17042022 was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the QC control limits for LCS/LCSD and MS/MSD 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 method blanks.

4.11.4. Field duplicate sample and its associated sample: Field sample MW36D042022 was identified as field duplicate of sample MW36D042022D. No Mercury was detected in the sample and associated field duplicate sample.

4.11.5. Raw data was submitted for all samples. Samples TMW52042022 (Lab ID#22D193-01) and TMW61042022 (Lab ID#22D193-12) were selected to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #22D193 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

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Final Data Validation Report

USACE Fort Wingate Depot Activity
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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of thirteen (13) water samples were collected on 04-20-22. EMAX Laboratories received the samples on 04-21-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Two samples from this sample delivery group, MW27042022(Lab ID#D205-01) and MW30042022 (Lab ID#D205-07) were designated as stage 3 deliverable. Raw data for these samples was compared to the reported summary tables for each method and went through comprehensive data validation review. Sample TMW15042022 (Lab ID#D205-04) was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank, LCS/LCSD and MS/MSD for each method were cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (13 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (9 samples)
EPA Method SW846 3535A/8330B: Nitroaromatics and Nitramines (9 samples)
EPA Method 8081B: Organochlorine Pesticides (7 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (5 sample)
EPA Method 8151A: Chlorinated herbicides (5 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (10 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (9 samples)

EPA Method 6850: Perchlorate (11 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (12 samples)

EPA Method 7470A: Mercury & Dissolved Mercury (12 samples)
EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate (12 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 # 22D205 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. Method SW3535A/8330B (Nitroaromatics and Nitramines) and method SW846 6850 (Perchlorate) was sub contracted by EMAX to GEL Laboratories in South Carolina.

A total of thirteen (13) water samples were collected on 04-20-22. EMAX Laboratories received the samples on 04-21-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the data was subjected to validation to the equivalent of stage 3.

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if presented

- Results and percent recoveries of Lab Control Samples (LCS/LCSD)
- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses and chromatograms for the sample/samples at Stage 3 deliverable and related QC samples.

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. Two field samples MW27042022 (EMAX ID #D205-01) and MW30042022 (EMAX ID #D205-07) from this sample delivery group was designated for stage 3 data review. Raw data for these samples was evaluated comprehensively. Sample TMW15042022
(Lab ID#D205-04) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for method blank, LCS/LCSD and MS/MSD was reviewed comprehensively. 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#22D205 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW27042022	22D205-01	04-20-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Anions by IC
TMW13042022	22D205-02	04-20-22	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW13042022D	22D205-03	04-20-22	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW15042022	22D205-04	04-20-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
MW37042022	22D205-05	04-20-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22D205 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW06042022	22D205-06	04-20-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Anions by IC
MW30042022	22D205-07	04-20-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Anions by IC
TMW35042022	22D205-08	04-20-22	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
MW18D042022	22D205-09	04-20-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22D205 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
MW35042022	22D205-10	04-20-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Anions by IC
MW35042022D	22D205-11	04-20-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Anions by IC
MW20042022	22D205-12	04-20-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Anions by IC
QC20042022TB8	22D205-13	04-20-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

Site Name: Fort Wingate, New Mexico

SDG#22D205 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW15042022MS	22D205-04M	04-20-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW15042022MSD	22D205-04S	04-20-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines 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
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20μg/L, (Benzidine=40μg/L)
	Nitroaromatics &Nitramines	SW3535A/8330B	1.25-5μg/L
	Nitroglycerine & PETN	SW3535A/8330B	2.5μg/L
	Chlorinated Herbicides	SW8151A	1μg/L, (MCPA=40μg/L)
	Organochlorine Pesticides	SW8081B	0.1μg/L Methoxychlor =1.0μg/L Toxaphene =2.0μg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1μg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100μg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5μg/L,1μg/L,20μg/L,100μg/L,200μg/L
	Dissolved Mercury/Mercury	SW7470A	0.5μg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2μg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements with qualifications if applied.

TABLE 2-2
Summary of Analytical Methods and Holding Time Requirements
USACE Wingate, New Mexico

ANALYSIS Method	MATRIX	HOLDING TIME REQUIREMENT	DATA QUALIFIED AS "J"
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	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 fifteen ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 0.9˚C and as high as 2.8˚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) =13/13X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =9/9X100=100%
Completeness (EPA Method 3535A/8330B: Explosives) =9/9X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =7/7X100=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) 10/10X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =9/9X100=100%
Completeness (EPA Method 6850: Perchlorate) =11/11X100=100%
Completeness (EPA Method 7470A: Mercury & Dissolved Mercury) =12/12X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =12/12X100=100%
Completeness (EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate) =12/12X100=100%

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

4.0 DATA VALIDATION

This data review covers thirteen water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **3535A/8330B** for Nitrtoaromatics and Nitramine by LC/MS/MS
EPA Method **6850** for Perchlorate by HPLC/MS/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 samples. thirteen water samples were collected on 04-20-22. Samples were analyzed on 04-28-22 and 04-29-22 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

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-05-22 and at the beginning of analysis shift on 04-28-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-05-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-05-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

Minimum average response factors for all the target compounds were within method’s recommended values, except for: Acetone (0.022), 2-Butanone (0.010), 4-Methyl-2-pentanone (0.086) and 2-Hexanone (0.062). 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 04-05-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-06-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standards were analyzed at the beginning and end of analysis shift on 04-28-22 and 04-29-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-28-22 (I)	Continuing cal. Response factors 04-29-22 (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 (04-28-22) I	%Deviation from Initial calibration (04-29-22) 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 target analytes listed in the table below for closing daily standards. This should not affect the data quality.

Target analyte	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration	
		(04-28-22) I	(04-29-22) II
Trichlorofluoromethane	≤ 20%	26.3*	√
2,2-Dichloropropane	≤ 20%	√	25.8*
2-Chloroethyl vinyl ether	≤ 20%	37.5*	43.6*
Carbon disulfide	≤ 20%	√	36.5*
4-Methyl-2-pentanone	≤ 20%	√	23.3*
Chloromethane	≤ 20%	√	35.6*
2-Hexanone	≤ 20%	√	22.6*

*Failed maximum 20% D

4.1.5. Quality Control samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW15042022 was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the full compound list for LCS/LCSD and MS/MSD. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

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

4.1.6. **Field duplicate sample** and its associated sample: Field sample TMW13042022 was identified as field duplicate of sample TMW13042022D and sample MW35042022 was identified as field duplicate of sample MW35042022D. No VOCs were detected in each of field samples or associated field duplicate samples, except traces of Acetone in sample MW35042022, as indicated in the table below:

Target analyte	MW35042022 (Lab ID #D205-10) µg/L	MW35042022D (Lab ID #D205-11) µg/L	**% RPD**
Acetone	U	8.9J	200

4.1.7. Raw data was submitted for all samples. Samples MW27042022 (Lab ID# 22D205-01) and MW30042022 (Lab ID# 22D205-07) were designated to be reviewed as stage 3 data deliverable. Raw data for these samples with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

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

4.2.1. Technical Holding Times

Holding time requirement was met for all samples. Nine water samples were collected on 04-20-22, extracted on 04-26-22 and were analyzed on 04-28-22 within required holding time.

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

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

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 04-28-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

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

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

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

√ denotes passing method acceptance limits

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

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

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

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

√ denotes passing method acceptance limits

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

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

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

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

4.2.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-28-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

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

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (04-28-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for 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 (04-28-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target analyte list except for the compound listed below in the opening continuing calibration standard:

Target Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (04-28-22) I	%Deviation from Initial calibration (04-28-22) II
Pyridine	≤ 20	21.9%*	21.4%*

*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 one set of LCS/LCSD and MS/MSD. Sample TMW115042022 was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Three compounds in LCS/LCSD and six target analytes in MS/MSD exceeded RPD limit of 20%.

Percent recoveries and percent RPDs for all the QC samples reported were mostly within the project acceptance limits, except for some compound as listed in the table below:

Target analyte	%LCS	%LCSD	%Recovery Limit	%RPD	%RPD Limit
Caprolactam	66	88	30-160	29*	≤ 20
Benzaldehyde	51	63	30-160	21*	≤ 20
1,2,4,5-Tetrachlorobenzene	47	58	35-121	21*	≤ 20
Target analyte	**%MS**	**%MSD**	%Recovery Limit	%RPD	%RPD Limit%
2,4-Dichlorophenol	70	55	47-121	22*	≤ 20
2-Chloronaphthalene	83	63	40-116	25*	≤ 20
2-Chlorophenol	58	44	38-117	25*	≤ 20
2-Nitrophenol	69	54	47-123	22*	≤ 20
Phenol	59	46*	50-130	23*	≤ 20
2,6-Dichlorophenol	68	54	50-118	22*	≤ 20

*Outside QC limit

Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision for these compounds.

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

Surrogate recoveries were all within the method's acceptable limits, except for surrogates in sample listed in the table below:

Surrogate	Surrogate Recovery Limit %	TMW06042022 (D205-06)
2-Fluorophenol	19-119	16%*
Phenol-D5	40-130	36%*

*Failed surrogate acceptance limits

4.2.6. **Field duplicate sample** and its associated sample: Field sample MW35042022 was identified as field duplicate of sample MW35042022D. No SVOCs were detected in each of field sample or associated field duplicate sample.

4.2.7. Raw data was submitted for all samples. Samples MW27042022 (Lab ID# 22D205-01) and MW30042022 (Lab ID# 22D205-07) 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.

4.3. 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. Seven water samples were collected on 04-20-22, extracted on 04-26-22 and analyzed on 05-03-22 and 05-04-22 within required holding time.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 03-11-22, 03-12-22 and before sample analysis on 05-03-22, and 05-04-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

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

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

4.3.3. Initial Calibration Verification and Continuing Calibration

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

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

A total of three continuing calibration standards were analyzed at 10-injections interval on 05-03-22, and 05-04-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A. Seven analytes, from opening daily standard run on 05-04-22 and almost all analytes in closing daily standard exceeded 20% limit in column B (all high biased). Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In all continuing calibration standards, one mid-point concentration of 20-40µg/L was injected.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked as 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. Results and recoveries of QC samples were confirmed with the reported raw data.

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

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Field sample MW35042022 was identified as field duplicate of sample MW35042022D. No pesticides were detected in each of field sample or associated field duplicate sample.

4.3.6. Raw data was submitted for all samples. Samples MW27042022 (Lab ID# 22D205-01) and MW30042022 (Lab ID# 22D205-07) 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 water samples requested for this method. Five water samples were collected on 04-20-22, extracted on 04-26-22, and analyzed on 04-27-22, within holding time requirement.

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 03-17-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-17-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-27-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

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

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

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

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample and its associated sample: Field sample MW35042022 was identified as field duplicate of sample MW35042022D. No Polychlorinated Biphenyls (PCBs) was detected in each of field sample or associated field duplicate sample.

4.4.6. Raw data was submitted for all samples. Samples MW27042022 (Lab ID# 22D205-01) and MW30042022 (Lab ID# 22D205-07) 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.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Five water samples were collected on 04-20-22, extracted on 04-25-22 and analyzed on 04-29-22 within holding time requirement.

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 04-28-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99922 (channel A) and 0.99976 (channel B) was used for MCPP.

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. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 04-28-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-29-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels.

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

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

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

4.5.5. Field duplicate sample and its associated sample: Field sample MW35042022 was identified as field duplicate of sample MW35042022D. No Chlorinated Herbicides was detected in each of field sample or associated field duplicate sample.

4.5.6. Raw data was submitted for all samples. Samples MW27042022 (Lab ID# 22D205-01) and MW30042022 (Lab ID# 22D205-07) 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.

4.6. Nitroaromatics by LC/MS/MS (EPA Method3535A/ 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 04-20-22, prepared on 04-27-22 and analyzed on 05-06-22, within holding time.

A High-Performance LC (HPLC) coupled with two Mass Detectors in tandem (LC/MS/MS) was used for analysis. Samples were sub-contracted to GEL Laboratories in South Carolina by EMAX Laboratory.

4.6.2. Initial Calibration

One initial calibration curve with seven concentration levels for each analyte was established on 05-05-22. Internal standard curve type was used for initial calibration and all the following analysis. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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) for all target analytes, except one analyte (p-Nitrotoluene). linear regression curve fit with correlation coefficient of 0.9978 was used for p-Nitrotoluene.

4.6.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 05-06-22. Percent recoveries were within required method limits (70-130% of the true value).

Two continuing calibration standards were analyzed at the start and end of analysis shift on 05-06-22. Percent recoveries were all within required method limits (80-120% of the true value) in the standards associated with sample analysis. However, Nitroglycerine exceeded the acceptance limits in the closing continuing calibration standard as listed in the table below:

Target analyte	05-06-2022 Daily St I	05-12-2022 Daily St II	**QC Limit%**
Nitroglycerine	--	127%*	80-120

*Exceeded QC limits

Associated samples were all non-detect for this analyte. Therefore, it does not affect the data quality.

The Initial calibration blanks and continuing calibration blanks were all non-detect.

4.6.4. Quality Control samples consisted of one method blank and one set of LCS. Sample TMW15042022 was designated to be spiked as MS/MSD, but the data was missing from data package with no comment on the case narrative. The entire explosive target lists were spiked and reported for LCS. Percent recoveries (%R) were within the QAPP established QC limits for all target analytes.

Result and recoveries of LCS was used for quality assurance. 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 (44-142%).

3,4-Dinitrotoluene was used as surrogate.

4.6.5 Field duplicate sample and its associated sample**:** Field sample MW35042022 was identified as field duplicate of sample MW35042022D. No explosives was detected in each of field sample or associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Samples MW27042022 and MW30042022 were designated 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.

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

4.7.1. 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 04-20-22. Samples were analyzed on 04-25-22 within holding time requirement.

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

4.7.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-07-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.7.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-25-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.7.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW35042022 was spiked as MS/MSD for this method. 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.

4.7.5. Field duplicate sample and its associated sample: Field sample MW35042022 was identified as field duplicate of sample MW35042022D. No TPH as Gasoline was detected in the field sample or associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Samples MW27042022(Lab ID#22D205-01) and MW30042022 (Lab ID#22D205-07) 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.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for nine water samples requested for this method. Water samples were collected on 04-20-22, extracted on 04-25-22 and analyzed on 04-27-22 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.

4.8.2. 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.

4.8.3. Initial 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%

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-27-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was spiked as MS/MSD for this method. 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.

4.8.5. Field duplicate sample and its associated sample: Field sample MW35042022 was identified as field duplicate of sample MW35042022D. No TPH as DRO was detected in the field sample or associated field duplicate sample.

4.8.6. Raw data was submitted for all samples. Samples MW27042022(Lab ID#22D205-01) and MW30042022 (Lab ID#22D205-07) 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.

4.9. Perchlorate by HPLC/MS/MS (EPA Method 6850 Modified)

4.9.1. 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 04-20-22. Samples were prepared on 05-02-22 and analyzed on 05-03-22 within holding time.

A High-Performance LC coupled with two Mass Detectors (HPLC/MS/MS) was used for analysis. The sample for this method was sub-contracted by EMAX Laboratory to GEL (General Engineering Laboratories) in South Carolina.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-30-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. Linear curve fit was used in the quantitation. The correlation coefficient of 0.99993 (Perchlorate) and 0.99992 (perchlorate 101) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.05 – 10 µg/L. All the QC requirements were satisfied.

Retention time for each isotope at each calibration level was within 0.2 minutes required by the method.

4.9.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-30-22. Percent recoveries were within required method limits (90-110% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of three daily standards were carried out on 05-03-22 bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 90-110% limit.

4.9.4. Quality Control samples consisted of one method blank, one LCS and one set of MS/MSD. Client sample MW27042022 was spiked and reported as MS/MSD. Sample TMW15042022 was designated to be spiked as MS/MSD on the chain of custody, but GEL spiked a different sample from the same sample delivery group. Perchlorate was spiked and reported for LCS and MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS and MS/MSD (84-119%). Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Interference check sample (ICS) was analyzed and results were within acceptable limit (80-120% of the true value).

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.

4.9.5. Field duplicate sample and its associated sample: Field samples TMW13042022 was identified as field duplicate of sample TMW13042022D and MW35042022 was identified as field duplicate of sample MW35042022D.

Results for Perchlorate are summarized in the table below for sample and sample duplicate:

Target analyte	TMW13042022 µg/L	TMW13042022D µg/L	**% RPD**
Perchlorate	0.059	0.062	4.96
	MW35042022 µg/L	MW35042022D µg/L	**% RPD**
Perchlorate	U	U	--

4.10.6. Raw data was submitted for all samples. Samples MW27042022 and MW30042022 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.

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

4.10.1. 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 04-20-22. Samples were prepared (digested) for both total and dissolved metals on 04-25-22. Samples and QC samples were analyzed on 05-04-22 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.

4.10.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-04-22.
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. Five levels of concentration were used for each initial calibration. 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 the 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.

4.10.3. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW15042022 was designated to be spiked and reported as MS/MSD for total and dissolved metals. 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 few metals as summarized in the table below. As a result, all the positive results will be qualified as estimated value "J" for these metals in the parent sample.

ANALYTE	Total Metals			Dissolved Metals		
	TMW15042022 MS%	TMW15042022 MSD%	QC Limit %	TMW15042022 MS%	TMW15042022 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	√	√	87-115
Calcium	√	√	87-118	√	√	87-118
Chromium	√	√	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	108	184*	85-118
Iron	77*	76*	87-118	√	√	87-118
Lead	√	√	88-115	√	√	88-115
Magnesium	√	√	83-118	√	√	83-118
Manganese	83*	84*	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	533*	300*	85-117	167*	300*	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. Samples TMW15042022 was used for serial dilution for total and dissolved metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis for both sets of data. 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.

4.10.4. Field duplicate sample and its associated sample: Field samples TMW13042022 was identified as field duplicate of sample TMW13042022D and sample MW35042022 was identified as field duplicate of sample MW35042022D. Results and %RPD for field sample and associated field duplicate sample are listed in the tables below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW13042022 (Lab ID#D205-02) µg/L	TMW13042022D (Lab ID#D205-03) µg/L	% RPD	TMW13042022 (Lab ID#D205-02) µg/L	TMW13042022D (Lab ID#D205-03) µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	17	17	<1	17	17	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	28000	27000	3.64	27000	27000	<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	5100	5100	<1	5100	5000	1.98
Manganese	U	U	--	U	U	--
Nickel	U	U	--	U	U	--
Potassium	340J	330J	2.98	360J	340J	5.71
Selenium	10	9.4	6.19	9.3	9.5	2.13
Silver	U	U	--	U	U	--
Sodium	580000	590000	1.71	580000	580000	<1
Thallium	U	U	--	U	U	--
Vanadium	U	U	--	U	U	--
Zinc	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	MW35042022 (Lab ID#D205-10) µg/L	MW35042022D (Lab ID#D205-11) µg/L	% RPD	MW35042022 (Lab ID#D205-10) µg/L	MW35042022D (Lab ID#D205-11) µg/L	% RPD
Aluminum	U	300J	200	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	9.6	14	37.3	11	10	9.53
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	19000	19000	<1	19000	19000	<1
Chromium	1.2J	0.53J	77.5	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	MW35042022 (Lab ID#D205-10) µg/L	MW35042022D (Lab ID#D205-11) µg/L	% RPD	MW35042022 (Lab ID#D205-10) µg/L	MW35042022D (Lab ID#D205-11) µg/L	% RPD
Iron	U	170J	200	U	U	--
Lead	U	U	--	U	U	--
Magnesium	2600	2700	3.77	2700	2700	<1
Manganese	36	42	15.4	35	36	2.82
Nickel	U	U	--	U	1.6J	200
Potassium	1100	1100	<1	1100	1100	<1
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	580000	770000	1.29	790000	780000	1.27
Thallium	U	U	--	U	U	--
Vanadium	1.3J	1.4J	7.41	1.4J	U	200
Zinc	U	U	--	U	U	--

4.10.5. Raw data was submitted for all samples. Samples MW27042022 (Lab ID# D205-01) and MW30042022 (Lab ID# D205-07) 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. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.11.1. 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 04-20-22. Samples were prepared (digested) on 05-03-22 and analyzed on 05-03-22 for Mercury and dissolved Mercury. All samples were preserved and filtered in the lab for dissolved Mercury analysis. Therefore, two sets of data were generated, one for Mercury and one for dissolved Mercury.

4.11.2. Initial and continuing calibrations: The instrument calibrations for this method were acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 05-03-22. The correlation coefficient of at least 0.999899 was calculated to show the linearity of each calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.11.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of one method blank one set of LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Sample TMW15042022 was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the QC control limits for LCS/LCSD and MS/MSD for both Mercury and dissolved Mercury. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.11.4. Field duplicate sample and its associated sample: Field samples TMW13042022 was identified as field duplicate of sample TMW13042022D and sample MW35042022 was identified as field duplicate of sample MW35042022D. No Mercury was detected in the field samples or associated field duplicate samples.

4.11.5. Raw data was submitted for all samples. Samples MW27042022 (Lab ID#22D205-01) and MW30042022 (Lab ID#22D205-07) 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.

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

4.12.1. Technical Holding Times: Holding time from sample collection to analysis was met for analysis of twelve water samples requested for this method. Water samples were collected on 04-20-22. Samples were analyzed on 04-21-22 for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride within the required 48-hour holding time. Samples were re-analyzed at higher dilutions for Chloride and Sulfate on 05-10-22 within 28-day holding times.

4.12.2. Initial and continuing calibration: Anions such as Chloride, Fluoride, Bromide, Nitrite, Nitrate, Orthophosphate and Sulfate were separated from water samples by Ion chromatography.

The separated anions in their acid form (very conductive) were measured by conductivity. They were identified on the basis of retention time as compared to reference standards.

Instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-04-22, and 05-04-22. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each anion. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 03-04-22 and 05-04-22. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 04-21-22 and 05-10-22. A total of nine continuing calibration standards were analyzed with analysis and re-analysis of all samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.12.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD, MS/MSD and sample/sample duplicate. Sample TMW15042022 was designated to be spiked as MS/MSD. The same sample was used for sample/sample duplicate analysis. A second set of QCs (MB and LCS/LCD) was analyzed with dilutions and/or re-analysis of samples for Chloride and Sulfate. Recoveries of LCS/LCSDs were within 90-110 % of spiked values for each anion. Results and recoveries of MS/MSD were within the QC limits for Nitrate, Nitrite, Orthophosphate and Sulfate. Remaining anions failed the required QC limits in MS/MSD as summarized in the table below:

Target analyte	TMW15042022MS	TMW15042022MSD	**QC Limit%**
Fluoride	118%*	118%*	82-112
Bromide	43%*	44*%	91-110
Chloride	121%*	114%*	87-111

*Outside QC limits

As a result, all the positive results will be qualified as estimated value "J" for these anions in the parent sample. Recoveries of sample/sample duplicate were all within acceptance limit, except Bromide, %RPD exceeded 20%limit (46%).

Percent RPDs were less than 20% for each set of LCS/LCSD for all anions.

4.12.4. Field duplicate sample and its associated sample: Field sample TMW13042022 was identified as field duplicate of sample TMW13042022D and sample MW35042022 was identified as field duplicate of sample MW35042022D. Results for each set of sample/sample duplicates are summarized in the tables below:

Anion	TMW13042022 (Lab ID #D205-02) mg/L	TMW13042022D (Lab ID #D205-03) mg/L	**% RPD**
Nitrate	4.0	4.0	<1
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.77	0.77	<1
Chloride	87	89	2.27
Fluoride	1.8	1.8	<1
Sulfate	350	360	2.82

Anion	MW35042022 (Lab ID #D205-10) mg/L	MW35042022D (Lab ID #D205-11) mg/L	**% RPD**
Nitrate	U	U	--
Nitrite	U	U	--
Orthophosphate	U	U	--
Bromide	0.79	0.79	<1
Chloride	320	320	<1
Fluoride	0.19	0.19	<1
Sulfate	1100	1000	9.52

4.12.5. Raw data was submitted for all requested field samples. Samples MW27042022 (Lab ID#22D205-01) and MW30042022 (Lab ID#22D205-07) were designated to be reviewed as stage 3 data deliverable. Raw data for these samples together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #22D205 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 #22D215 Analytical Data Package

Publication Date: 08-09-2022

By

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of ten (10) water samples were collected on 04-21-22. EMAX Laboratories received the samples on 04-22-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples was submitted for the requested analytical method. Sample TMW21042022 (Lab ID#D215-03) was designated to be reviewed as stage 3 deliverable. Raw data for this sample was compared to the reported summary tables and went through comprehensive data validation. No sample was designated to be spiked as MS/MSD. Recoveries and results of LCS/LCSD was used to evaluate accuracy and precision. Raw data for method blanks and LCS/LCSDs were cross checked with the corresponding summary table results.

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

EPA Method 9056A: Anions by IC (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 required analytical holding times were met for all anions. The deviations, if any, are discussed in Section 4.0 for this method.

The SDG # 22D215 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 04-21-22. EMAX Laboratories received the samples on 04-22-22.

2.1 Data Reporting

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

EMAX Laboratories provided the following information in one data package:

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

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

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for analytical method requested on the chain of custody were submitted for each sample. Sample TMW21042022 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. No sample was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blanks with each set of LCS/LCSD was reviewed. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);
- Technical holding times were confirmed for all samples with 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;
- LCS/LCSD results were evaluated; and
- Method blank results as well as surrogate recoveries were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#22D215 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
MW31042022	D215-01	04-21-22	S3VM	Anions by IC,
TMW25042022	D215-02	04-21-22	S3VM	Anions by IC,
TMW21042022	D215-03	04-21-22	S3VM	Anions by IC,
BGMW13S042022	D215-04	04-21-22	S3VM	Anions by IC,
TMW32042022	D215-05	04-21-22	S3VM	Anions by IC,
BGMW13D042022	D215-06	04-21-22	S3VM	Anions by IC,
TMW38042022	D215-07	04-21-22	S3VM	Anions by IC,
BGMW13D042022D	D215-08	04-21-2	S3VM	Anions by IC,
TMW47042022	D215-09	04-21-22	S3VM	Anions by IC,
MW22D042022	D215-10	04-21-22	S3VM	Anions by IC,

TABLE 2-1

Summary of Analytical Parameters
USACE Wingate, New Mexico

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

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

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

TABLE 2-2

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

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

2.2.3 Laboratory and Field Blanks

The objective of laboratory and field blanks is to determine the presence and extent of contamination resulting from laboratory or field activities. Blanks reported here included method blank only. The result of analysis of method blank is discussed in Section 4.0 for this method. Samples were transported in one ice preserved cooler and was stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as 5.4˚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:

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

Where:

RPD = Relative percent difference

R_1 = Result of the first duplicate or measured sample concentration

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

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

3.2.2 Accuracy

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

3.2.3 Completeness

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

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

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

Completeness (EPA Method 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, 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 ten (10) water samples were collected on 04-21-22. Samples were analyzed on 04-22-22 within the required 48-hour holding time for Nitrate, Nitrite and Orthophosphate. Samples were analyzed on 05-11-22 and 05-16-22 for Sulfate and Chloride within 28-day holding time. Analysis was within 28-day holding time for Bromide and Fluoride.

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

Instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-04-22 and 05-04-22. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each anion. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 03-04-22 and 05-04-22. Recoveries were all within

90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 04-22-22, 05-11-22 and 05-16-22. A total of eleven continuing calibration standards were analyzed with analysis and re-analysis of all samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

4.12.3. Quality Control Samples consisted of one method blank and set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. A second set of MB and LCS/LCSD was analyzed with Chloride and Sulfate analysis on 05-09-22. Recoveries of LCS/LCSDs were within 90-110 % of spiked values for each anion.

Percent RPDs were less than 20% for each set of LCS/LCSD for all anions.

4.12.4. Field duplicate sample and its associated sample: Sample BGMW13D042022 was identified as field duplicate of BGMW13D042022D. Results for sample/sample duplicate is summarized in the table below:

Anion	BGMW13D042022 (Lab ID #D177-08) mg/L	BGMW13D042022D (Lab ID #D177-10) mg/L	**% RPD**
Nitrate	U	U	--
Nitrite	U	U	--
Orthophosphate	0.47J	0.53	12.0
Bromide	0.46	0.45	2.20
Chloride	98	91	7.41
Fluoride	1.2	1.2	<1
Sulfate	62	67	7.75

4.12.5. Raw data was submitted for all requested field samples. Sample TMW21042022 (Lab ID#22D215-03) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #22D215 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 #22D227 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 April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of seven (7) water samples were collected on 04-22-22. EMAX Laboratories received the samples on 04-23-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. One sample from this sample delivery group, TMW40D042022(Lab ID#D227-06) 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 review. No sample was designated to be spiked as MS/MSD on the chain of custody. 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 cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (7 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (6 samples)
EPA Method SW846 3535A/8330B: Nitroaromatics and Nitramines (6 samples)
EPA Method 8081B: Organochlorine Pesticides (6 samples)
EPA Method 8082A: Polychlorinated Biphenyls; PCBs (2 sample)
EPA Method 8151A: Chlorinated herbicides (2 sample)
EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (3 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (2 samples)

EPA Method 6850: Perchlorate (6 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (6 samples)

EPA Method 7470A: Mercury & Dissolved Mercury (6 samples)
EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate (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 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 # 22D227 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. Method SW3535A/8330B (Nitroaromatics and Nitramines) and method SW846 6850 (Perchlorate) was sub contracted by EMAX to GEL Laboratories in South Carolina.

A total of seven (7) water samples were collected on 04-22-22. EMAX Laboratories received the samples on 04-23-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the data was subjected to validation to the equivalent of stage 3.

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if presented

- 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 TMW40D042022 (EMAX ID #D227-06) 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 comprehensively. 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#22D227 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW38042022	22D227-01	04-22-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Anions by IC
TMW49042022	22D227-02	04-22-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW39D042022	22D227-03	04-22-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
MW39042022	22D227-04	04-22-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#22D227 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW48042022	22D227-05	04-22-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW40D042022	22D227-06	04-22-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
QC22042022TB10	22D227-07	04-22-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

TABLE 2-1

Summary of Analytical Parameters
USACE Wingate, New Mexico

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW3535A/8330B	1.25-5µg/L
	Nitroglycerine & PETN	SW3535A/8330B	2.5µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements with qualifications if applied.

TABLE 2-2
Summary of Analytical Methods and Holding Time Requirements
USACE Wingate, New Mexico

ANALYSIS Method	MATRIX	HOLDING TIME REQUIREMENT	DATA QUALIFIED AS "J"
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	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 nine ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 0.7˚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:

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

Completeness (EPA Method 5030B/8260C: VOCs) =7/7X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =6/6X100=100%
Completeness (EPA Method 3535A/8330B: Explosives) =6/6X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =6/6X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =2/2X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =2/2X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 3/3X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =2/2X100=100%
Completeness (EPA Method 6850: Perchlorate) =6/6X100=100%
Completeness (EPA Method 7470A: Mercury & Dissolved Mercury) =6/6X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =6/6X100=100%
Completeness (EPA Method SW9056A: Nitrate-N, Nitrite, Orthophosphate, Bromide, Chloride, Fluoride and Sulfate) =6/6X100=100%

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

4.0 DATA VALIDATION

This data review covers seven water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **3535A/8330B** for Nitrtoaromatics and Nitramine by LC/MS/MS
EPA Method **6850** for Perchlorate by HPLC/MS/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 samples. Seven water samples were collected on 04-22-22. Samples were analyzed on 05-04-22 and 05-05-22 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

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-28-22 and at the beginning of analysis shift on 05-04-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-28-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-28-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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) 04-28-22
Acetone	0.9987

Minimum average response factors for all the target compounds were within method's recommended values, except for: Acetone (0.048) and 2-Butanone (0.069). 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 04-28-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-29-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standards were analyzed at the beginning and end of analysis shift on 05-04-22 and 05-05-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 05-04-22 (I)	Continuing cal. Response factors 05-05-22 (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 (05-04-22) I	%Deviation from Initial calibration (05-05-22) 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 target analytes listed in the table below for closing daily standards. This should not affect the data quality.

Target analyte	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration	
		(05-04-22) I	(05-05-22) II
Vinyl acetate	≤ 20%	23*	√
2-Butanone	≤ 20%	23.2	√
2,2-Dichloropropane	≤ 20%	√	27.8*

*Failed maximum 20% D

4.1.5. Quality Control samples reported consisted of one method blank and one set of LCS/LCSD. No sample was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs were within the project acceptance limits for the full compound list for LCS/LCSD. 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 collected with this sample delivery group.

4.1.7. Raw data was submitted for all samples. Sample TMW40D042022 (Lab ID# 22D227-06) 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 Toluene (0.11Jµg/L) was detected in trip blank. However, it was not reported in ant field sample.

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

4.2.1. Technical Holding Times

Holding time requirement was met for all samples. Six water samples were collected on 04-22-22, extracted on 04-28-22 and were analyzed on 05-02-22 within required holding time.

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

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

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 05-02-22. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group. Initial calibration curves were generated on 03-07-22 and 03-16-22. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

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

√ denotes passing method acceptance limits

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

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

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

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

√ denotes passing method acceptance limits

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

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

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

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

4.2.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 05-02-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

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

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (05-02-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for 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 (05-02-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target analyte list except for the compound listed below in the opening continuing calibration standard:

Target Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (05-02-22) I	%Deviation from Initial calibration (05-02-22) II
2,4-Dinitrophenol	≤ 20	√	27.5%*

*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. Quite a number of analytes (thirteen, mostly phenol compounds) in LCS/LCSD exceeded RPD limit of 20%.

Percent recoveries reported were mostly within the project acceptance limits.

Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision.

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 TMW40D042022 (Lab ID# 22D227-06) 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. Six water samples were collected on 04-22-22, extracted on 04-29-22 and analyzed on 05-03-22, 05-04-22 and 05-07-22 within required holding time.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 03-11-22, 03-12-22 and before sample analysis on 05-03-22, 05-04-22 and 05-07-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

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

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

4.3.3. Initial Calibration Verification and Continuing Calibration

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

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

A total of seven continuing calibration standards were analyzed at 10-injections interval on 05-03-22, 05-04-22 and 05-07-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A. Seven analytes, from opening daily standard and almost all analytes in closing daily standard run on 05-04-22 exceeded 20% limit in column B (all high biased). Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In all continuing calibration standards, one mid-point concentration of 20-40µg/L was injected.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked as 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.

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

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

Surrogate recoveries were all within the method QC acceptance limits.

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

4.3.6. Raw data was submitted for all samples. Sample TMW40D042022 (Lab ID# 22D227-06) 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.

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

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Two water samples were collected on 04-22-22, extracted on 04-29-22, and analyzed on 05-03-22, within holding time requirement.

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 03-17-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-17-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-03-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

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

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

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

Surrogate recoveries were all within the method QC acceptance limits.

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

4.4.6. Raw data was submitted for all samples. Sample MW38042022 (Lab ID# 22D227-01) 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.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Two water samples were collected on 04-22-22, extracted on 04-26-22 and analyzed on04-29-22 and 05-02-22 within holding time requirement.

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 04-28-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99922 (channel A) and 0.99976 (channel B) was used for MCPP.

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. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 04-28-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-29-22 and 05-02-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels.

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

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

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

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

4.5.6. Raw data was submitted for all samples. Sample MW38042022 (Lab ID# 22D227-01) 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.

4.6. Nitroaromatics by LC/MS/MS (EPA Method3535A/ 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 04-22-22, prepared on 04-29-22 and analyzed on 05-14-22, within holding time.

A High-Performance LC (HPLC) coupled with two Mass Detectors in tandem (LC/MS/MS) was used for analysis. Samples were sub-contracted to GEL Laboratories in South Carolina by EMAX Laboratory.

4.6.2. Initial Calibration

One initial calibration curve with seven concentration levels for each analyte was established on 05-12-22. Internal standard curve type was used for initial calibration and all the following analysis. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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) for all target analytes, except two. 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) 05-12-22**
PETN	0.9971
RDX	0.9961

4.6.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 05-12-22. Percent recoveries were within required method limits (70-130% of the true value).

Three continuing calibration standards were analyzed at the start and end of analysis shift on 05-14-22. Percent recoveries were all within required method limits (80-120% of the true value) in the three standards associated with the analytical shift. However, RDX exceeded the acceptance limits in the mid continuing calibration standard as listed in the table below:

Target analyte	05-14-2022 Daily St I	05-14-2022 Daily St II	05-14-2022 Daily St III	**QC Limit%**
RDX	√	124%*	√	80-120

*Exceeded QC limits

Associated samples were all non-detect for this analyte. Therefore, it does not affect the data quality.

The Initial calibration blanks and continuing calibration blanks were all non-detect.

4.6.4. Quality Control samples consisted of one method blank and one LCS. The entire explosive target lists were spiked and reported for LCS. Percent recoveries (%R) were within the QAPP established QC limits for all target analytes except for HMX as summarized in the table below:

Target analyte	1205078876 LCS%	**QC Limit%**
HMX	124*	62-112

*Exceeded QC limits

Since the recovery is biased high and the target analyte was not detected in the associated samples, the data will not be affected.

Result and recoveries of LCS was used for quality assurance. 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 (44-142%).

3,4-Dinitrotoluene was used as surrogate.

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

4.6.6. Raw data was submitted for all samples. Sample TMW40D042022 was designated as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.7.1. 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 04-22-22. Samples were analyzed on 04-25-22 and 04-26-22 within holding time requirement.

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

4.7.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-07-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.7.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-25-22 and 04-26-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.7.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD for this method. 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.

4.7.5. 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 MW38042022(Lab ID#22D227-01) was selected to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for two water samples requested for this method. Water samples were collected on 04-22-22, extracted on 04-28-22 and analyzed on 05-02-22 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.

4.8.2. 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.

4.8.3. Initial 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%

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-02-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was spiked as MS/MSD for this method. 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.

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

4.8.6. Raw data was submitted for all samples. Sample MW38042022(Lab ID#22D227-01) 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.

4.9. Perchlorate by HPLC/MS/MS (EPA Method 6850 Modified)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six samples requested for this method. Water samples were collected on 04-22-22. Samples were prepared on 05-02-22 and analyzed on 05-04-22 and 05-05-22 within holding time.

A High-Performance LC coupled with two Mass Detectors (HPLC/MS/MS) was used for analysis. The sample for this method was sub-contracted by EMAX Laboratory to GEL (General Engineering Laboratories) in South Carolina.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 05-04-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. Least square Linear curve fit was used in the quantitation. The correlation coefficient of 0.99985 (Perchlorate) and 0.99979 (perchlorate 101) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.05 – 10 µg/L. All the QC requirements were satisfied.

Retention time for each isotope at each calibration level was within 0.2 minutes required by the method.

4.9.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 05-04-22. Percent recoveries were within required method limits (90-110% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of six daily standards were carried out on 05-04-22 and 05-05-22 bracketing the analyses and re-analysis of samples and all the QC samples. Recoveries of continuing calibration standards were within 90-110% limit.

4.9.4. Quality Control samples consisted of one method blank, one LCS and one set of MS/MSD. A client sample TMW58042022 was spiked and reported as MS/MSD. This sample belongs to a different sample delivery group (SDG#22D120). Associated samples were all batched together. Perchlorate was spiked and reported for LCS and MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS and MS/MSD (84-119%). Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Interference check sample (ICS) was analyzed and results were within acceptable limit (80-120% of the true value).

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.

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

4.10.6. Raw data was submitted for all samples. Sample TMW40D042022 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.

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

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six water samples requested for this method. Water samples were collected on 04-22-22. Samples were prepared (digested) for both total and dissolved metals on 04-26-22. Samples and QC samples were analyzed on 05-05-22 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.

4.10.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-05-22.
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. Five levels of concentration were used for each initial calibration. 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 the 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.

4.10.3. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for total metals. Sample MW38042022 was spiked and reported as MS/MSD for total metals. QC for dissolved metals included one method blank and one set of LCS/LCSD 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 for few metals as summarized in the table below. As a result, all the positive results will be qualified as estimated value "J" for these metals in the parent sample for total metals.

ANALYTE	Total Metals			Dissolved Metals		
	MW38042022 MS%	MW38042022 MSD%	QC Limit %	NA MS%	NA MSD%	QC Limit%
Aluminum	109	169*	84-117	NA	NA	84-117
Antimony	√	√	85-117	NA	NA	85-117
Arsenic	√	√	84-116	NA	NA	84-116
Barium	110	167*	86-114	NA	NA	86-114
Beryllium	√	√	83-121	NA	NA	83-121
Cadmium	√	√	87-115	NA	NA	87-115
Calcium	97	53*	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	110	140*	87-118	NA	NA	87-118
Lead	√	√	88-115	NA	NA	88-115
Magnesium	80*	290*	83-118	NA	NA	83-118
Manganese	√	√	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	-67*	0.00*	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 MW38042022 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 and dissolved metals.

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

4.10.5. Raw data was submitted for all samples. Sample TMW40D042022 (Lab ID# D227-06) 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. MERCURY & Dissolved Mercury by COLD VAPOR: EPA Method 7470A

4.11.1. 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 04-22-22. Samples were prepared (digested) on 04-29-22 and analyzed on 04-29-22 for Mercury and dissolved Mercury. All samples were preserved and filtered in the lab for dissolved Mercury analysis. Therefore, two sets of data were generated, one for Mercury and one for dissolved Mercury.

4.11.2. Initial and continuing calibrations: The instrument calibrations for this method were acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 04-29-22. The correlation coefficient of at least 0.999557 was calculated to show the linearity of each calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.11.3. Quality Control: The data for all the QC samples were within acceptable control limits. 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 QC control limits for LCS/LCSD for both Mercury and dissolved Mercury. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

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

4.11.5. Raw data was submitted for all samples. Sample TMW40D042022 (Lab ID#22D227-06) 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.

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

4.12.1. 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 04-22-22. Samples were analyzed on 04-23-22 for Nitrate, Nitrite, Orthophosphate, Bromide and Fluoride within the required 48-hour holding time. Samples were -analyzed at higher dilutions for Chloride and Sulfate on 05-05-22 within 28-day holding times.

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

Instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-04-22, and 05-04-22. Linear curve type with correlation coefficient of at least 0.999 for each anion was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each anion. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 03-04-22 and 05-04-22. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 04-23-22 and 05-05-22. A total of seven continuing calibration standards were analyzed with analysis and re-analysis of all samples. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed with reference standard. It was within the assigned QC limit for each anion.

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

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

4.12.5. Raw data was submitted for all requested field samples. Sample TMW40D042022 (Lab ID#22D227-06) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #22D227 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 #22D229 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 April 2022 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of eleven (11) water samples were collected on 04-21-22. EMAX Laboratories received the samples on 04-23-22. Data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. One sample from this sample delivery group, TMW21042022(Lab ID#D229-04) 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 review. No sample was designated to be spiked as MS/MSD on the chain of custody. 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 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 (8 samples)
EPA Method SW846 3535A/8330B: Nitroaromatics and Nitramines (10 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) (7 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (6 samples)
EPA Method 6850: Perchlorate (9 samples)
EPA Method SW6020A: Dissolved and total Metals by ICP-MS (10 samples)
EPA Method 7470A: Mercury & Dissolved Mercury (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 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 # 22D229 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. Method SW3535A/8330B (Nitroaromatics and Nitramines) and method SW846 6850 (Perchlorate) was sub contracted by EMAX to GEL Laboratories in South Carolina.

A total of eleven (11) water samples were collected on 04-21-22. EMAX Laboratories received the samples on 04-23-22.

2.1 Data Reporting

The data was delivered in one package as stage 2b and stage 3 deliverables. 10% of the data was subjected to validation to the equivalent of stage 3.

EMAX Laboratories provided the following information in one data package:

- Sample identification number;
- Date of sample collection;
- Sample matrix type;
- Analysis method;
- Target lists and results of analysis;
- Limit of Detection (LOD);
- Limit of Quantitation (LOQ);
- Laboratory qualifiers and qualifier definitions;
- Copies of sample logs and chain-of-custody logs;
- Sample preparation logs (with the sample extraction dates);
- Sample Analysis logs (Instrument injection logs with sample analysis dates);
- Results and percent recoveries of Matrix Spike Samples (MS/MSD), if presented

- 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 TMW21042022 (EMAX ID #D229-04) 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 comprehensively. 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#22D229 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW31042022	22D229-01	04-21-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
BGMW13S042022	22D229-02	04-21-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
TMW25042022	22D229-03	04-21-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS
TMW21042022	22D229-04	04-21-22	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO
MW32042022	22D229-05	04-21-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#22D229 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
BGMW13D042022	22D229-06	04-21-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
TMW38042022	22D229-07	04-21-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
BGMW13D042022D	22D229-08	04-21-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH Gasoline; TPH as DRO
TMW47042022	22D229-09	04-21-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#22D229 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
MW22042022	22D229-10	04-21-22	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls
QC21042022TB9	22D227-11	04-21-22	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

TABLE 2-1

Summary of Analytical Parameters
USACE Wingate, New Mexico

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW3535A/8330B	1.25-5µg/L
	Nitroglycerine & PETN	SW3535A/8330B	2.5µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.2µg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

Technical holding times are defined as the maximum time allowed between sample collection, extraction and analysis. Collection to extraction and extraction-to-analysis (40-day) was within the holding time requirement for semi-volatile organic methods. Extraction-to-analysis was within the method's holding time requirement with metals and inorganic methods. Table 2-2 presents the summary of holding time requirements with qualifications if applied.

TABLE 2-2
Summary of Analytical Methods and Holding Time Requirements
USACE Wingate, New Mexico

ANALYSIS Method	**MATRIX**	**HOLDING TIME REQUIREMENT**	**DATA QUALIFIED AS "J"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	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 twelve ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 0.7˚C and as high as 3.4˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

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

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

Where:

RPD = Relative percent difference

R_1 = Result of the first duplicate or measured sample concentration

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

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

3.2.2 Accuracy

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

3.2.3 Completeness

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

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

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

Completeness (EPA Method 5030B/8260C: VOCs) =11/11X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =8/8X100=100%
Completeness (EPA Method 3535A/8330B: Explosives) =10/10X100=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) 7/7X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =6/6X100=100%
Completeness (EPA Method 6850: Perchlorate) =9/9X100=100%
Completeness (EPA Method 7470A: Mercury & Dissolved Mercury) =10/10X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =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 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 **3535A/8330B** for Nitrtoaromatics and Nitramine by LC/MS/MS
EPA Method **6850** for Perchlorate by HPLC/MS/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Mercury & 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. Eleven water samples were collected on 04-21-22. Samples were analyzed on 05-03-22 and 05-04-22 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

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-28-22 and at the beginning of each analysis shift on 05-03-22 and 05-04-22. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-28-22. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-28-22
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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) 04-28-22
Acetone	0.9987

Minimum average response factors for all the target compounds were within method's recommended values, except for: Acetone (0.048) and 2-Butanone (0.069). 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 04-28-22
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-29-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds.

Continuing calibration check standards were analyzed at the beginning and end of each analysis shift on 05-03-22 and 05-04-22. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria. Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 05-03-22 (I&II)	Continuing cal. Response factors 05-04-22 I&(II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance

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 (05-03-22) I&II	%Deviation from Initial calibration (05-04-22) I&II
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for target analytes listed in the table below for closing daily standards. This should not affect the data quality.

Target analyte	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration		%Deviation from Initial calibration	
		(05-03-22) I	(05-03-22) II	(05-04-22) I	(05-04-22) II
Tetrahydrofuran	≤ 20%	26.8*	√	24.4*	√
2-Butanone	≤ 20%	√	21.7*	√	√
Bromochloromethane	≤ 20%	√	24.6*	√	√
1,2-Dichloroethane	≤ 20%	√	21.4*	√	√
4-Methyl-2-pentanone	≤ 20%	√	21.9*	√	√
Bromoform	≤ 20%	√	27*	√	√

*Failed maximum 20% D

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

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

4.1.6. **Field duplicate sample** and its associated sample. Field sample BGMW13D042022 was identified as field duplicate of sample BGMW13D042022D. No VOCs was detected in field sample or associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample TMW21042022 (Lab ID# 22D229-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

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

4.2.1. Technical Holding Times

Holding time requirement was met for all samples. Eight water samples were collected on 04-21-22, extracted on 04-28-22 and were analyzed on 05-02-22 within required holding time.

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

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

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 03-07-22, 03-16-22 and at the beginning of analysis shift on 05-02-22. It passed all the method assigned criteria. Tailing of

Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

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

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

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

√ denotes passing method acceptance limits

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

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

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

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

√ denotes passing method acceptance limits

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

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

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

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

4.2.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-08-22 and 03-16-22. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 05-02-22. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

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

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (05-02-22) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for 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 (05-02-22) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (05-02-22) I& II
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target analyte list except for the compound listed below in the closing continuing calibration standard:

Target Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (05-02-22) I	%Deviation from Initial calibration (05-02-22) II
2,4-Dichlorophenol	≤ 20	√	27.5%*

*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. Quite a number of analytes (thirteen, mostly phenol compounds) in LCS/LCSD exceeded RPD limit of 20%. Percent recoveries reported were mostly within the project acceptance limits.

Results and recoveries of LCS/LCSD was used to evaluate accuracy and precision.

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 surrogates in sample listed in the table below:

Surrogate	Surrogate Recovery Limit %	TMW47042022 (D229-09)
2-Fluorophenol	19-119	0.0%*
Phenol-D5	40-130	10%*
2,4,6-Tribromophenol	43-140	32%*

*Failed surrogate acceptance limits

4.2.6. **Field duplicate sample** and its associated sample: Field sample BGMW13D042022 was identified as field duplicate of sample BGMW13D042022D. No SVOCs were detected in each of field sample or associated field duplicate sample.

4.2.7. Raw data was submitted for all samples. Sample TMW21042022 (Lab ID# 22D229-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all water samples requested for this method. Eight water samples were collected on 04-21-22, extracted on 04-27-22 and analyzed on 05-02-22, 05-03-22 and 05-07-22 within required holding time.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 03-11-22, 03-12-22 and before sample analysis on 05-02-22, 05-03-22 and 05-07-22. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

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

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

4.3.3. Initial Calibration Verification and Continuing Calibration

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

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

A total of six continuing calibration standards were analyzed at 10-injections interval on 05-02-22, 05-03-22 and 05-07-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A. Almost all analytes, from daily standards run on 05-03-22 (three total) exceeded 20% limit in column B (all high biased). Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In all continuing calibration standards, one mid-point concentration of 20-40µg/L was injected.

4.3.4. Quality Control samples consisted of method blank and one set of LCS/LCSD. No sample was designated to be spiked as 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. Results and recoveries of QC samples were confirmed with the reported raw data.

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

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Field sample BGMW13D042022 was identified as field duplicate of sample BGMW13D042022D. No pesticides were detected in each of field sample or associated field duplicate sample.

4.3.6. Raw data was submitted for all samples. Sample TMW21042022 (Lab ID# 22D229-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.4. 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 04-21-22, extracted on 04-27-22, and analyzed on 04-28-22, within holding time requirement.

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 03-17-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-17-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-28-22 and 04-29-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

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

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

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

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample and its associated sample: Field sample BGMW13D042022 was identified as field duplicate of sample BGMW13D042022D. No Polychlorinated Biphenyls (PCBs) were detected in each of field sample or associated field duplicate sample.

4.4.6. Raw data was submitted for all samples. Sample MW31042022 (Lab ID# 22D229-01) 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.

4.5. 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 04-21-22, extracted on 04-26-22 and analyzed on 04-29-22 and 05-02-22 within holding time requirement.

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 04-28-22. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99922 (channel A) and 0.99976 (channel B) was used for MCPP.

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. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 04-28-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-29-22 and 05-02-22, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels.

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

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

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

4.5.5. Field duplicate sample and its associated sample: Field sample BGMW13D042022 was identified as field duplicate of sample BGMW13D042022D. No Chlorinated Herbicides were detected in each of field sample or associated field duplicate sample.

4.5.6. Raw data was submitted for all samples. Sample MW31042022 (Lab ID# 22D229-01) 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.

4.6. Nitroaromatics by LC/MS/MS (EPA Method3535A/ 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 04-21-22, prepared on 04-28-22 and analyzed on 05-08-22, within holding time.

A High-Performance LC (HPLC) coupled with two Mass Detectors in tandem (LC/MS/MS) was used for analysis. Samples were sub-contracted to GEL Laboratories in South Carolina by EMAX Laboratory.

4.6.2. Initial Calibration

One initial calibration curve with seven concentration levels for each analyte was established on 05-07-22. Internal standard curve type was used for initial calibration and all the following analysis. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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) for all target analytes.

4.6.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 05-07-22. Percent recoveries were within required method limits (70-130% of the true value).

Two continuing calibration standards were analyzed at the start and end of analysis shift on 05-08-22. Percent recoveries were all within required method limits (80-120% of the true value) in the two standards associated with the analytical shift.

The Initial calibration blanks and continuing calibration blanks were all non-detect.

4.6.4. Quality Control samples consisted of one method blank and one set of LCS. A set of MS/MSD from non-client sample (marked as non-SDG) was presented with this data package. The entire explosive target lists were spiked and reported for LCS. Percent recoveries (%R) were within the QAPP established QC limits for all target analyte. Result and recoveries of LCS was used for quality assurance. 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 (44-142%) except for sample BGMW13D042022D. 3,4-Dinitrotoluene was used as surrogate. Surrogate recovery was reported as 0.0% for this sample, therefore all analytes were qualified as “UQ” for this sample. No trace of surrogate was detected in the corresponding chromatogram. It looks like surrogate was not spiked. According to case narrative, there was not enough water left for re-extraction of the sample.

4.6.5 Field duplicate sample and its associated sample**:** Field sample BGMW13D042022 was identified as field duplicate of sample BGMW13D042022D. No explosives were detected in each of field sample or associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample TMW21042022 was designated as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.7.1. 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 04-21-22. Samples were analyzed on 04-25-22 and 04-26-22 within holding time requirement.

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

4.7.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 04-07-22. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.7.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-07-22. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-25-22 and 04-26-22, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.7.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD for this method. 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.

4.7.5. Field duplicate sample and its associated sample: Field sample BGMW13D042022 was identified as field duplicate of sample BGMW13D042022D. No TPH as Gasoline was detected in the field sample or associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample TMW21042022(Lab ID#22D229-04) was 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.

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

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 04-21-22, extracted on 04-28-22 and analyzed on 05-02-22 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.

4.8.2. 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.

4.8.3. Initial 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%

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-02-22 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD for this method. 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.

4.8.5. Field duplicate sample and its associated sample: Field sample BGMW13D042022 was identified as field duplicate of sample BGMW13D042022D. Trace of TPH as DRO was detected in the field duplicate sample as indicated in the table below.

Target analyte	BGMW13D042022 (Lab ID #D229-06) mg/L	BGMW13D042022D (Lab ID #D229-08) mg/L	**% RPD**
TPH as DRO	U	0.12J	200

4.8.6. Raw data was submitted for all samples. Sample TMW21042022(Lab ID#22D229-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.9. Perchlorate by HPLC/MS/MS (EPA Method 6850 Modified)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for nine samples requested for this method. Water samples were collected on 04-21-22. Samples were prepared on 04-29-22 and analyzed on 05-03-22 and 05-05-22 within holding time.

A High-Performance LC coupled with two Mass Detectors (HPLC/MS/MS) was used for analysis. The sample for this method was sub-contracted by EMAX Laboratory to GEL (General Engineering Laboratories) in South Carolina.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-30-22. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. Least square Linear curve fit was used in the quantitation. The correlation coefficient of 0.99993 (Perchlorate) and 0.99992 (perchlorate 101) was calculated to show the linearity of each curve. A second set of initial calibration was run on 05-04-22. The correlation coefficient of 0.99979 (Perchlorate) and 0.99985 (perchlorate 101) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.05 – 10 µg/L. All the QC requirements were satisfied.

Retention time for each isotope at each calibration level was within 0.2 minutes required by the method.

4.9.3. Initial Calibration Verification and Continuing Calibration

Each initial calibration was verified by a second source standard (ICV) on 04-30-22 and 05-04-22. Percent recoveries were within required method limits (90-110% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of six daily standards were carried out on 05-03-22 and 05-05-22 bracketing the analyses and re-analysis of samples and all the QC samples. Recoveries of continuing calibration standards were within 90-110% limit.

4.9.4. Quality Control samples consisted of one method blank, one LCS and one set of MS/MSD. A client sample BGMW07042022 was spiked and reported as MS/MSD. This sample belongs to a different sample delivery group (SDG#22D109-01). Associated samples were all batched together. Perchlorate was spiked and reported for LCS and MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS and MS/MSD (84-119%). Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Interference check sample (ICS) was analyzed and results were within acceptable limit (80-120% of the true value).

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.

4.9.5. Field duplicate sample and its associated sample: Field sample BGMW13D042022 was identified as field duplicate of sample BGMW13D042022D. No Perchlorate was detected in field sample or associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample TMW21042022 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.

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

4.10.1. 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 04-21-22. Samples were prepared (digested) for both total and dissolved metals on 04-26-22. Samples and QC samples were analyzed on 05-05-22 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.

4.10.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-05-22.

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. Five levels of concentration were used for each initial calibration. 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 the 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.

4.10.3. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for total metals. Sample MW31042022 was spiked and reported as MS/MSD for total metals. QC for dissolved metals included one method blank and one set of LCS/LCSD 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 for few metals as summarized in the table below. As a result, all the positive results will be qualified as estimated value "J" for these metals in the parent sample for total metals.

ANALYTE	Total Metals			Dissolved Metals		
	MW31042022 MS%	MW31042022 MSD%	**QC Limit %**	NA MS%	NA MSD%	**QC Limit%**
Aluminum	√	√	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	100	133*	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	175*	90	87-118	NA	NA	87-118
Lead	√	√	88-115	NA	NA	88-115
Magnesium	√	√	83-118	NA	NA	83-118
Manganese	√	√	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	-667*	-330*	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 MW31042022 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 and dissolved metals.

4.10.4. Field duplicate sample and its associated sample: Field sample BGMW13D042022 was identified as field duplicate of sample BGMW13D042022D. Results and %RPD for field sample and associated field duplicate sample are listed in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	BGMW13D042022 µg/L	BGMW13042022D µg/L	% RPD	BGMW13D042022 µg/L	BGMW13D042022D µg/L	% RPD
Aluminum	200J	280J	33.3	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	260	260	<1	240	240	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	17000	18000	5.71	14000	15000	6.90
Chromium	18	16	11.8	1.9J	2.8J	38.3
Cobalt	0.54J	U	200	U	U	--
Copper	U	U	--	15	13	14.3
Iron	610J	620J	1.63	500J	150J	108
Lead	0.31J	0.34J	9.23	U	U	--
Magnesium	4300	4400	2.3	4100	4100	<1
Manganese	150	150	<1	120	120	<1
Nickel	19	17	11.1	2.2J	2.4J	8.70
Potassium	850J	850J	<1	800J	840J	4.88
Selenium	U	U	--	9.3	9.5	2.13
Silver	U	U	--	U	U	--
Sodium	290000	290000	<1	290000	290000	<1
Thallium	U	U	--	U	U	--
Vanadium	1.9J	2.5J	27.3	1.3J	1.7J	26.7
Zinc	U	U	--	U	U	--

4.10.5. Raw data was submitted for all samples. Sample TMW21042022 (Lab ID# D229-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. 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 04-21-22. Samples were prepared (digested) on 05-05-22 and analyzed on 05-05-22 for Mercury and dissolved Mercury. All samples were preserved and filtered in the lab for dissolved Mercury analysis. Therefore, two sets of data were generated, one for Mercury and one for dissolved Mercury.

4.11.2. Initial and continuing calibrations: The instrument calibrations for this method were acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 05-05-22. The correlation coefficient of at least 0.999757 was calculated to show the linearity of each calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.11.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of one method blank, one set of LCS/LCSD and MS/MSD for Mercury. Sample BGMW13S042022 was selected to be spiked as MS/MSD for Mercury. QC for dissolved Mercury included one method blank and one set of LCS/LCSD only. Percent recoveries and % differences were within the QC control limits for LCS/LCSD for both Mercury and dissolved Mercury. Results for MS/MSD were within QC acceptable limits. Sample BGMW13S042022 was also used for serial dilution for Mercury. Serial dilution analysis, (at 5-fold dilution) was within 10% difference of the initial analysis. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.11.4. Field duplicate sample and its associated sample: Field sample TMW21042022 was identified as field duplicate of sample TMW21042022D. No Mercury was detected in the field sample or associated field duplicate sample.

4.11.5. Raw data was submitted for all samples. Sample TMW21042022 (Lab ID#22D229-04) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #22D229 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

6.0 REFERENCES

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

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

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.