Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #21D152 Analytical Data Package

Publication Date: 06-20-21

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

Five (5) water samples were collected on 04-12-21. EMAX Laboratories received the samples on 04-13-21. Data was delivered in one package as stage 2b and stage 3 deliverable. Data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples was submitted for the requested analytical method. Sample TMW36042021 (Lab ID#D152-03) was designated as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation. No sample was designated to be spiked as MS/MSD on the chain of custody. Therefore, results and recoveries of LCS/LCSD was used to evaluate both accuracy and precision for Nitrate and Nitrite.

Stage 2b data validation examined quality assurance/quality control (QA/QC) elements such as holding time, preparation 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 9056A: Anions by IC (5 samples)

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

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

The SDG # 21D152 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, 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.

Five (5) water samples were collected on 04-12-21. EMAX Laboratories received the samples on 04-13-21.

2.1 Data Reporting

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

EMAX Laboratories provided the following information in one data package:

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

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

Data validation was performed by initial review of the analytical reports and QA/QC results and recoveries using summary tables. Then, selected analytical reports including QA/QC information was cross checked with raw data. The analysis sequence log for the method was examined. Overall review assessed the effects of QA/QC results on the data usability. The review included such parameters as holding times, LODs/LOQs, initial and continuing calibration method requirements, lab control sample/lab control sample duplicate (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 analytical method requested on the chain of custody were submitted for each sample. Sample TMW36042021 was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample together with QC samples were evaluated comprehensively at stage 2b and stage 3 data validation review. 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

- MS/MSD results, if requested, were evaluated;
- LCSs results were evaluated; and
- Method blank results as well as surrogate recoveries were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#21D152 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation Stage**	**Requested Methods of Analysis**
BGMW07042021	D152.01	04-12-21	S3VM	Anions by IC,
TMW18042021	D152.02	04-12-21	S3VM	Anions by IC,
TMW36042021	D152.03	04-12-21	S3VM	Anions by IC,
BGMW03042021	D152.04	04-12-21	S3VM	Anions by IC,
TMW44042021	D152.05	04-12-21	S3VM	Anions by IC,

TABLE 2-1

Summary of Analytical Parameters

USACE Wingate, New Mexico

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

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

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 ice preserved 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	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 4.4˚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:

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

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: Nitrate and Nitrite) =5/5X100=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 five 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 Nitrate and Nitrite by IC

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

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

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

The following Reason codes were applied in the report:

M3	MS/MSD and/or LCS/LCSD percent recovery infraction with low bias

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

4.1. Method SW9056A: Nitrate-N and Nitrite

4.1.1. Technical Holding Times: Holding time from sample collection to analysis was met for five (5) water samples requested for this method. Water samples were collected on 04-12-21 and analyzed on 04-13-21, within 48-hour holding time.

4.1.2. Initial and continuing calibration:

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

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-04-21. Linear curve type with correlation coefficient of at least 0.999199 (for Nitrate) and 0.999763 (for Nitrite) was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture at mid-point (4.0mg/L) was used to verify the linearity of initial calibration on 03-04-2021. Recoveries were within 90-110% of initial value. Continuing calibration standards at 10-injections interval were analyzed on 04-13-21. A total of six continuing calibration standards were analyzed with sample and re-analysis of some anions. In all continuing calibrations 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 was established and confirmed in each 24-hour. It was within the assigned QC limit for each anion.

4.1.3. Quality Control Samples consisted of method blank and LCS/LCSD only. No sample was assigned to be spiked as MS/MSD or sample/sample duplicate. Recoveries of LCS/LCSD were within expected QC limits (88-111 % of spiked values). Percent RPDs were less than 15%.

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

4.1.5. Sample TMW36042021 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 #21D152 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 #21D168 Analytical Data Package

Publication Date: 06-25-21

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of thirteen (13) water samples were collected on 04-13-21. EMAX Laboratories received the samples on 04-14-21. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample TMW58042021(Lab ID# D168-06) was assigned as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation. Sample TMW26042021(Lab ID# D168-12) was designated to be spiked as MS/MSD on the chain of custody. Raw data for this sample together with method blank and LCS/LCSD for each method were also cross checked with the corresponding summary table results.

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

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

EPA Method 7470: Mercury & Dissolved Mercury (12 samples)
EPA Method SW9056A: Nitrate-N and Nitrite (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 # 21D168 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. However, one sample (MW32042021 at DLX100) was analyzed for Nitrate three hours past 48 hours required holding time. Qualifications are discussed in section 4.13.5. 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.

Thirteen (13) water samples were collected on 04-13-21. EMAX Laboratories received the samples on April 14, 2021.

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 TMW58042021 (EMAX ID #D168-06) from this sample delivery group was designated for stage 3 data review. Raw data for this sample was evaluated comprehensively. Sample TMW26042021 (EMAX ID #D168-12) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with other QC samples in this report LCS/LCSD was also reviewed in detail. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#21D168 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW37042021	21D168-01	04-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
MW23042021	21D168-02	04-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW46042021	21D168-03	04-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Anions by IC
QC13042021TB2	21D168-04	04-13-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
MW32042021	21D168-05	04-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D168 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW58042021	D168-06	04-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
SMW01042021	D168-07	04-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
SMW01042021D	D168-08	04-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
BGMW12042021	D168-09	04-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D168 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW01042021	D168-10	04-13-21	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Anions by IC
BGMW08042021	D168-11	04-13-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW26042021	D168-12	04-13-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
MW02042021	D168-13	04-13-21	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D168 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW26042021MS	D168-12M	04-13-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW26042021MSD	D168-12S	04-13-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC

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

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L
	Perchlorate	SW6850	0.5µg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

TABLE 2-2

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

ANALYSIS Method	**MATRIX**	**HOLDING TIME REQUIREMENT**	**DATA QUALIFIED AS "J"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	None. Holding times were met
Anions by IC	Water	Analysis 48 hours from collection	Holding times were met for all except one sample*
Dissolved and Total Mercury	Water	Analysis within 6 Months	None. Holding times were met

* Dilution for sample MW32042021 (at X100) was analyzed 3 hours past HT for Nitrate; This sample was initially analyzed with dilution X10 within holding time. Therefore, reported result will be quailed as estimated value "J".

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 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:

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

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) =13/13X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =9/9X100=100%
Completeness (EPA Method 8330B: Explosives) =12/12X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =12/12X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =9/9X100=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 7470: Dissolved and Total Mercury) =12/12X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =12/12X100=100%
Completeness (EPA Method SW9056A: Nitrate-N and Nitrite) =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 **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor
EPA **Method 9056A** for Nitrate and Nitrite 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 04-13-21. Samples were analyzed on 04-16-21, and 04-19-21 within method's requirement for holding time. (Water samples were preserved with hydrochloric acid).

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

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

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 02-23-21 and at the beginning of each analysis shift on 04-16-21 and 04-19-21. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 02-23-21. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 02-23-21
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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) 02-23-21
Tetrahydrofuran	0.9978

Average response factors for all target compounds were within method's recommended values, except for: Acetone (0.036) and 2-Butanone (0.062).

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 02-23-21
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 02-24-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds except for Tetrahydrofuran (27.1%).

Continuing calibration check standard was analyzed at the beginning and end of each analysis shift on 04-16-21 and 04-19-21. Prior to each continuing calibration, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-16-21 (I& II)	Continuing cal. Response factors 04-19-21 (I& II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-16-21) I&II	%Deviation from Initial calibration (04-19-21) I&II
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for Dibromochloromethane (23.1%) in the closing daily standard run on 04-19-21. This should not affect data quality.

4.1.5. Quality Control: The QC samples reported consisted of two method blanks, two sets of LCS/LCSD and MS/MSD. Sample TMW26042021 (lab ID #D168-12) was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for MS/MSD and LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list.

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

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

4.1.6. **Field duplicate sample** and its associated sample: Sample SMW01042021 was identified as field duplicate of SMW01042021D. Both sample and corresponding field duplicate sample were reported as non-detected for all volatile compound list.

4.1.7. Raw data was submitted for all samples. Sample TMW58042021 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 Bromoform, Bromodichloromethane and Dibromochloromethane were detected in trip blank. These compounds were not reported in any field samples.

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 04-13-21, extracted on 04-14-21 and were analyzed on 04-16-21, 04-19-21 and 04-26-21 within required holding time.

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

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

4.2.2. Tuning criteria

Performance of the instrument was checked by injection of a tune check standard (DFTPP: Decafluorotriphenylphosphine) prior to initial calibration on 09-21-20, 09-23-20 and at the beginning of each analysis shift on 04-16-21, 04-19-21 and 04-26-21. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

Initial calibration curve was generated on 09-21-20 and 10-27-20. 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 09-21-20
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 15%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 09-21-20
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) 09-21-20&10-27-20
Benzoic acid	0.9996
2,4-Dinitrophenol	0.9989
4,6-Dinitro-2-Methylphenol	0.9973
Benzidine	0.9983
Dinoseb	0.9998
1,3,5-Trinitrobenzene	0.9992
3,3-dimethylbenzidine	0.9987
Di-n-Octylphthalate	0.9998

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 09-23-20 and 10-27-20. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds except Acenaphthene (25.4%). Continuing calibration check standards were analyzed at the beginning and at the end of each analysis shift on 04-16-21, 04-19-21 and 04-26-21. Prior to each continuing calibration, 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 (04-16-21) I&II	Continuing cal. Response factors (04-19-21) I& II	Continuing cal. Response factors (04-26-21) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√	√
2,4-Dinitrophenol	≥ 0.01	√	√	√
4-Nitrophenol	≥ 0.01	√	√	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (4-16-21) I&II	Accepted Deviation from Initial calibration (04-19-21) I&II	Accepted Deviation from Initial calibration (04-26-21) I& II
Phenol	≤ 20	√	√	√
1,4-Dichlorobenzene	≤ 20	√	√	√
2-Nitrophenol	≤ 20	√	√	√
2,4-Dichlorophenol	≤ 20	√	√	√
Hexachlorobutadiene	≤ 20	√	√	√
4-Chloro-3-methylphenol	≤ 20	√	√	√
2,4,6-Trichlorophenol	≤ 20	√	√	√
Acenaphthene	≤ 20	√	√	√
N-Nitrosodiphenylamine	≤ 20	√	√	√
Pentachlorophenol	≤ 20	√	√	√
Fluoranthene	≤ 20	√	√	√
Di-n-Octylphthalate	≤ 20	√	√	√
Benzo(a)pyrene	≤ 20	√	√	√

√ denotes passing method acceptance limits

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

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (04-16-21) II	%Deviation from Initial calibration (04-19-21) II	%Deviation from Initial calibration (04-26-21) II
Anilin	≤ 20	27.9*	--	--
2,4-Dinitrophenol	≤ 20	23.2*	--	23.2
3,3-Dimethylbenzidine	≤ 20	30.3*	25.9*	--
Di-n-octylphthalate	≤ 20	--	21.5*	--
Hexachlorocyclopentadiene	≤ 20	--	--	22.00*
Benzo€pyrene	≤ 20	--	--	25.4*

*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: The QC samples reported consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits for the entire compound list in LCS/LCSD. Recoveries of LCS/LCSD was used to evaluate accuracy and precision for this method.

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

4.2.6. **Field duplicate sample** and its associated sample: Sample SMW01042021 was identified as field duplicate of SMW01042021D. Both sample and corresponding field duplicate sample were reported as non-detected for the entire Semi-Volatile Compound list.

4.2.7. Raw data was submitted for all samples. Sample TMW58042021 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. Nine (9) water samples were collected on 04-13-21, extracted on 04-16-21 and analyzed on 04-19-21.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 02-17-21 and before sample analysis on 04-19-21. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 02-17-21 and 02-18-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 02-18-21 and 02-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

A total of three continuing calibration standards were analyzed at 10-injections interval on 04-19-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A and B. In the three continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only.

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

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

Surrogate recoveries were all within the method QC acceptance limits.

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

4.3.6. Raw data was submitted for all samples. Sample TMW58042021 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.

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 04-13-21, extracted on 04-16-21, and analyzed on 04-20-21.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-10-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-10-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors were injected at single point for identification only.

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

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

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

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

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample: No field duplicate sample was collected for this method.

4.4.6. Raw data was submitted for all samples. Sample TMW58042021 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 04-13-21, extracted on 04-19-21 and analyzed on 04-20-21.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 02-09-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list.

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

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 02-09-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

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

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

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

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

4.5.6. Raw data was submitted for all samples. Sample TMW58042021 with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 04-13-21, extracted on 04-19-21 and analyzed on 04-21-21 and 04-22-21, within holding time.

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

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 10-26-20. Confirmation (Kinetex-Biphenyl column) was calibrated on 01-20-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

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

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for both columns on 10-27-20 and 01-20-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of five continuing calibration standards (both primary and confirmation columns) were analyzed on 04-21-21, 04-22-21 and 04-26-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD. Sample TMW26042021 was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD and MS/MSD. However, one compound (3,5-DNA) was missed in the spiking mix, but it was included in the initial and continuing calibrations. EMAX Laboratory acknowledged this in the case narrative for this method. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.6.5 Field duplicate sample and its associated sample**:** Sample SMW01042021 was identified as field duplicate of sample SMW01042021D. No explosive target compound was detected in sample and field duplicate sample.

4.6.6. Raw data was submitted for all samples. Samples TMW58042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation column. One compound, 3,5-DNA was missed from the QC list for this method. This compound was among initial calibration and continuing calibration standards, and was also reported in the raw data.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 04-13-21, extracted on 04-19-21 and analyzed on 04-21-21.

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

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 10-27-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (Less than 15 %.)

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

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-21-21, bracketing the analyses of samples and all the QC samples.

Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD. Sample TMW26042021 was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.7.5. Field duplicate sample and its associated sample**:** Sample SMW01042021 was identified as field duplicate of sample SMW01042021D. No explosive target compound was detected in sample and field duplicate sample.

4.7.6. Raw data was submitted for all samples. Samples TMW58042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Seven (7) water samples were collected on 04-13-21. Samples were analyzed on 04-14-21 and 04-15-21 within holding time requirement.

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

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 03-16-21. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 03-16-21. 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 04-14-21 and 04-15-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

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

Surrogate recoveries were all within the method QC acceptance limits.

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

4.8.6. Raw data was submitted for all samples. Sample TMW58042021 was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water sample requested for this method. Six (6) water samples were collected on 04-13-21, extracted on 04-14-21 and analyzed on 04-17-21.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-01-20. Calibration factor (area for each compound/concentration) was used to quantify diesel range hydrocarbons (TPH as DRO). A second set of initial calibration curve was generated for lighter TPHs (Jet Fuel) and heavier TPHs (motor oil). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 %.)

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

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-01-20. 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 04-17-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated for MS/MSD. Percent recoveries (%R) of LCS/LCSD were within the

QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

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

4.9.6. Raw data was submitted for all samples. Sample TMW58042021 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. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Twelve (12) water samples were collected on 04-13-21. Samples were analyzed on 04-15-21.

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

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-09-21. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9996 (Perchlorate ion 83) and 0.9997 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

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

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-09-21. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-15-21, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW26042021 was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets of LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.10.5. Field duplicate sample and its associated sample: Sample SMW01042021 was identified as field duplicate of SMW01042021D. No perchlorate was detected in each sample and associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Samples TMW58042021 was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve water samples requested for this method. Water samples were collected on 04-13-21. Samples were prepared (digested) for both total and dissolved metals on 04-21-21. Samples and QC samples were analyzed on 05-03-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-03-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method's acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

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

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

4.11.3. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW26042021 was spiked as MS/MSD. Recoveries of LCS/LCSD were all within the acceptance limit of 80-120% for both total and dissolved metals. Recoveries of MS/MSD were mostly acceptable except the Sodium as listed in the table below. As a result, all the positive results for parent sample will be qualified as estimated value "J". This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	TMW26042021 MS%	TMW26042021 MSD%	QC Limit%	TMW26042021 MS%	TMW26042021 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		
	TMW26042021 MS%	TMW26042021 MSD%	**QC Limit%**	TMW26042021 MS%	TMW26042021 MSD%	**QC Limit%**
Cadmium	√	√	87-115	√	√	87-115
Calcium	√	√	87-118	√	√	87-118
Chromium	√	√	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	√	√	85-118
Iron	√	√	87-118	√	√	87-118
Lead	√	√	88-115	√	√	88-115
Magnesium	√	√	83-118	√	√	83-118
Manganese	√	√	87-115	√	√	87-115
Nickel	√	√	85-117	√	√	85-117
Potassium	√	√	85-115	√	√	85-115
Selenium	√	√	80-120	√	√	80-120
Silver	√	√	85-116	√	√	85-116
Sodium	-567*	-200*	85-117	-467*	-433*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

Method blank results for metals were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard. Sample TMW26042021 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. The same sample was used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total and dissolved metals.

4.11.4. Field duplicate sample and its associated sample: Sample SMW01042021 was identified as field duplicate of SMW01042021D. Results for sample/sample duplicated is summarized in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	SMW01042021 µg/L	SMW01042021D µg/L	**% RPD**	SMW01042021 µg/L	SMW01042021D µg/L	**% RPD**
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	SMW01042021 µg/L	SMW01042021D µg/L	% RPD	SMW01042021 µg/L	SMW01042021D µg/L	% RPD
Arsenic	2.5J	2.4J	4.08	2.3J	2.1J	9.1
Barium	47	46	2.15	25	25	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	22000	22000	<1	22000	22000	<1
Chromium	U	U	--	U	U	--
Cobalt	1.2J	1.2J	<1	U	U	--
Copper	U	U	--	U	5.2J	200
Iron	U	U	--	U	U	--
Lead	U	U	--	U	U	--
Magnesium	7500	7600	1.32	7700	7500	2.63
Manganese	860	860	<1	41	75	58.6
Nickel	3.0J	2.8J	6.89	U	U	--
Potassium	U	U	--	U	U	--
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	1000000	1000000	<1	1000000	1000000	<1
Thallium	U	U	--	U	U	--
Vanadium	6.2J	6.4J	<1	5.3J	5.3J	<1
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Sample TMW58042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.12. MERCURY by COLD VAPOR (Dissolved and total): EPA Method 7470A

4.12.1. 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 04-13-21. Samples were prepared (digested) and analyzed on 04-22-21 for Mercury. Samples were digested and analyzed on 04-23-21 for dissolved Mercury. All samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of each analysis day on 04-22-21 and 04-23-21. The correlation coefficient of at least 0.999 was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of method blank, LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Sample TMW26042021 was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW26042021 was also used for serial dilution. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample SMW01042021 was identified as field duplicate of SMW01042021D. No Mercury was detected in sample and corresponding field duplicate sample for total and dissolved Mercury.

4.12.5. Raw data was submitted for all samples. Samples TMW58042021 was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from raw data, agreed with all the results reported in data summary reports.

4.13. Method SW9056A: Nitrate-N and Nitrite

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for initial analysis of water samples requested for this method. Twelve (12) water samples were collected on 04-13-21. Samples were analyzed on 04-14-21 and 04-15-21 within the required 48-hour holding time. All initial analysis were within 48 hours holding time. However, sample

MW32042021 at dilution X100, was analyzed three hours past required 48-hour holding time for Nitrate. Since exceedance for holding time is less than 2XHT, the positive result for Nitrate would be qualified as estimated value ("J") for this sample.

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

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-04-21. Linear curve type with correlation coefficient of at least 0.999 was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of initial calibration on 03-04-21. Recoveries were all within 90-110% of initial true value. Continuing Calibration standards at 10-injections interval were analyzed on 04-14-21 and 04-15-21. A total of eight continuing calibration standards were analyzed with re-analysis of some anions. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed in each 24-hour. It was within the assigned QC limit for each anion.

4.13.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD, MS/MSD and sample/ sample duplicate analysis. Sample TMW26042021 was spiked as MS/MSD and also used for sample duplicate analysis. Recoveries of LCS/LCSD and MS/MSD were within 90-110 % of spiked values for Nitrate and Nitrite. Percent RPDs were less than 20% for LCS/LCSD, MS/MSD and sample/sample duplicate.

4.13.4. Field duplicate sample: and its associated sample: Sample SMW01042021 was identified as field duplicate of SMW01042021D. No Nitrate or Nitrite was detected in each sample and associated field duplicate sample.

4.13.5. Raw data was submitted for all requested field samples. Sample TMW58042021 was designated to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met. As mentioned in section 4.13.1, sample MW32042021 at dilution X100, was analyzed three hours past 48-hour required holding time for Nitrate. Since exceedance is less than 2XHT requirement, the positive result for Nitrate would be qualified as estimated value ("J") for this sample.

5.0 CONCLUSION

SDG #21D168 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

6.0 REFERENCES

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

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

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

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

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

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

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

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #21D169 Analytical Data Package

Publication Date: 06-25-21

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of nine (9) water samples were collected on 04-12-21. EMAX Laboratories received the samples on 04-14-21. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample TMW36042021(Lab ID# D169-03) was assigned as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation. No sample was designated to be spiked as MS/MSD on the chain of custody. Therefore, results and recoveries of LCS/LCSD was used to evaluate both accuracy and precision. Raw data for method blank and LCS/LCSD for each method were also cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (9 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (8 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (8 samples)
EPA Method SW8332: Nitroglycerine and PETN (8 samples)
EPA Method 8081B: Organochlorine Pesticides (4 samples)
EPA Method 8082: Polychlorinated Biphenyls; PCBs (1 sample)
EPA Method 8151A: Chlorinated herbicides (1 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (1 samples)
EPA Method 6850: Perchlorate (8 samples)
EPA Method SW6020A: Dissolved and total Metals by ICP-MS (8 samples)
EPA Method 7470: 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 # 21D169 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.

Nine (9) water samples were collected on 04-12-21. EMAX Laboratories received the samples on April 14, 2021.

2.1 Data Reporting

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

EMAX Laboratories provided the following information in one data package:

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

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

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. One field sample TMW36042021 (EMAX ID #D169-03) 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. Therefore, recoveries of LCA/LCSD were used for evaluation of accuracy and precision in each method. Raw data for LCS/LCSD and method blanks were also reviewed in detail. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#21D169 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW18042021	21D169-01	04-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
QC12042021TB1	21D169-02	04-12-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW36042021	21D169-03	04-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
BGMW07042021	21D169-04	04-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls
TMW44042021	21D169-05	04-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850

Site Name: Fort Wingate, New Mexico

SDG#21D169 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
BGMW03042021	D169-06	04-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
TMW19042021	D169-07	04-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850
TMW16042021	D169-08	04-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850
TMW16042021D	D169-09	04-12-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850

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

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L
	Perchlorate	SW6850	0.5µg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

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

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

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) =9/9X100=100%

Completeness (EPA Method 3520B/8270D: SVOCs) =8/8X100=100%

Completeness (EPA Method 8330B: Explosives) =8/8X100=100%

Completeness (EPA Method 8332: Nitroglycerine & PETN) =8/8X100=100%

Completeness (EPA Method 8081B: Organochlorine pesticides) =4/4X100=100%

Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =1/1X100=100%

Completeness (EPA Method 8151B: Chlorinated Herbicides) =1/1X100=100%

Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 1/1X100=100%

Completeness (EPA Method 6850: Perchlorate) =8/8X100=100%

Completeness (EPA Method 7470: Dissolved and Total 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 **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor

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 04-12-21. Samples were analyzed on 04-19-21 within method's requirement for holding time. (Water samples were preserved with hydrochloric acid).

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

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

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 02-23-21 and at the beginning of analysis shift on 04-19-21. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 02-23-21. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 02-23-21
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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) 02-23-21
Tetrahydrofuran	0.9978

Average response factors for all target compounds were within method's recommended values, except for: Acetone (0.036) and 2-Butanone (0.062).

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 02-23-21
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

The initial calibration was verified by a second source standard on 02-24-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds except for Tetrahydrofuran (27.1%).

Continuing calibration check standard was analyzed at the beginning and end of analysis shift on 04-19-21. Prior to opening 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 04-19-21 (I)	Continuing cal. Response factors 04-19-21 (II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-19-21) I	%Deviation from Initial calibration (04-19-21) II
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for Tetrahydrofuran (27.1%) in the opening daily standard and Dibromochloromethane (23.1%) in closing daily check standard. This should not affect data quality.

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

4.1.6. **Field duplicate sample** and its associated sample: Sample TMW16042021 was identified as field duplicate of TMW16042021D. Both sample and corresponding field duplicate sample were reported as non-detected for all Volatile Organic Compound list.

4.1.7. Raw data was submitted for all samples. Sample TMW36042021 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 Bromoform, Bromodichloromethane and Dibromochloromethane were detected in the trip blank. These compounds were not detected in any of the field samples.

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

4.2.1. Technical Holding Times

Holding time requirement was met for all samples. eight water samples were collected on 04-12-21, extracted on 04-19-21 and were analyzed on 04-21-21 and 04-22-21.

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 03-18-21 and at the beginning of each analysis shift on 04-21-21 and 04-22-21. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

Initial calibration curve was generated on 03-18-21 and 03-19-21. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-18-21
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 15%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-18-21
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 03-18-21
Benzoic acid	0.9985
2,4-Dinitrophenol	0.9990
4,6-Dinitro-2-Methylphenol	0.9982
Benzidine	0.9985
Dinoseb	0.9993
3,3-dimethylbenzidine	0.9987

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 03-19-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of each analysis shift on 04-21-21 and 04-22-21. Prior to each continuing calibration, 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 (04-21-21) I& II	Continuing cal. Response factors (04-22-21) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√
2,4-Dinitrophenol	≥ 0.01	√	√
4-Nitrophenol	≥ 0.01	√	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (04-21-21) I&II	Accepted Deviation from Initial calibration (04-22-21) I& II
Phenol	≤ 20	√	√
1,4-Dichlorobenzene	≤ 20	√	√
2-Nitrophenol	≤ 20	√	√
2,4-Dichlorophenol	≤ 20	√	√
Hexachlorobutadiene	≤ 20	√	√
4-Chloro-3-methylphenol	≤ 20	√	√
2,4,6-Trichlorophenol	≤ 20	√	√
Acenaphthene	≤ 20	√	√

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (04-21-21) I&II	Accepted Deviation from Initial calibration (04-22-21) I& II
N-Nitrosodiphenylamine	≤ 20	√	√
Pentachlorophenol	≤ 20	√	√
Fluoranthene	≤ 20	√	√
Di-n-Octylphthalate	≤ 20	√	√
Benzo(a)pyrene	≤ 20	√	√

√ denotes passing method acceptance limits

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

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (04-21-21) II	%Deviation from Initial calibration (04-22-21) II
Bis(2Chloroisopropylether)	≤ 20	22.0*	26.4
2,4-Dinitrophenol	≤ 20	26.5*	--
Di-n-Octylphthalate	≤ 20	21.0*	25.6*
4-Nitroaniline	≤ 20	21.2*	--

*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: The QC samples reported consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits for the entire compound list in LCS/LCSD. Recoveries of LCS/LCSD was used to evaluate accuracy and precision for this method.

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

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

4.2.6. **Field duplicate sample** and its associated sample: Sample TMW16042021 was identified as field duplicate of TMW16042021D. Both sample and corresponding field duplicate sample were reported as non-detected for the entire Semi-Volatiles Compound list.

4.2.7. Raw data was submitted for all samples. Sample TMW36042021 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 (8) water samples were collected on 04-12-21, extracted on 04-16-21 and analyzed on 04-19-21.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 02-17-21 and before sample analysis on 04-19-21. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 02-17-21 and 02-18-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was also generated for Toxaphene for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 02-18-21 and 02-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

A total of four continuing calibration standards were analyzed at 10-injections interval on 04-19-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for target compounds from channels A. Almost all compounds exceeded the maximum 20% limit in the closing daily standard for confirmation column. All the results were reported from the primary column. Therefore, this should not affect the data quality.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In the two 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 only. 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 within established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

Surrogate recoveries were all within the method QC acceptance limits.

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

4.3.6. Raw data was submitted for all samples. Sample TMW36042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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 sample requested for this method. One water sample was collected on 04-12-21, extracted on 04-16-21, and analyzed on 04-20-21.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-10-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-10-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors were injected at single point for identification only.

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

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

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

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

Surrogate recoveries were all within the method QC acceptance limits.

4.4.5. Field duplicate sample: No field duplicate sample was collected for this method.

4.4.6. Sample BGMW07042021 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 sample requested for this method. One water sample was collected on 04-12-21, extracted on 04-19-21 and analyzed on 04-20-21.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 02-09-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list.

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

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 02-09-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

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

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

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

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

4.5.6. Sample BGMW07042021 with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight (8) water samples requested for this method. Water samples were collected on 04-12-21, extracted on 04-19-21 and analyzed on 04-21-21 and 04-22-21, within holding time.

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

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 10-26-20. Since no explosives were detected in any of the field samples, calibration data for confirmation column was not submitted in the data package. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

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

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of four continuing calibration standards were analyzed on 04-21-21and 04-22-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD. However, one compound (3,5-DNA) was missed in the spiking mix, but it was included in the initial and continuing calibrations. EMAX Laboratory acknowledged this in the case narrative for this method. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.6.5 Field duplicate sample and its associated sample**:** Sample TMW16042021 was identified as field duplicate of sample TMW16042021D. No explosive target compound was detected in sample or field duplicate sample.

4.6.6. Raw data was submitted for all samples. Samples TMW36042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. One compound, 3,5-DNA was missed from the QC list for this method. This compound was among initial calibration and continuing calibration standards, and was also reported in the raw data.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight (8) water samples requested for this method. Water samples were collected on

04-12-21, extracted on 04-19-21 and analyzed on 04-21-21.

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

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 10-27-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (Less than 15 %.)

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

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of four continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-21-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

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

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

4.7.5. Field duplicate sample and its associated sample**:** Sample TMW16042021 was identified as field duplicate of sample TMW16042021D. No explosive target compound was detected in each sample and corresponding field duplicate sample.

4.7.6. Raw data was submitted for all samples. Samples TMW36042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water sample requested for this method. One (1) water sample were collected on 04-12-21. Sample was extracted and analyzed on 04-14-21 and 04-15-21 within holding time requirement.

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

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 03-16-21. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 03-16-21. 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 04-14-21 and 04-15-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

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

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

4.8.6. Sample QC12042021TB1 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 (EPA Method 6850)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Eight (8) water samples were collected on 04-12-21. Samples were analyzed on 04-15-21.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-09-21. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion

($Cl^{18}O4^-$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9996 (Perchlorate ion 83) and 0.9997 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

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

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-09-21. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-15-21, 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 and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established QC limits for both sets of LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.9.5. Field duplicate sample and its associated sample: Sample TMW16042021 was identified as field duplicate of TMW16042021D. No perchlorate was detected in each sample and associated field duplicate sample.

4.9.6. Raw data was submitted for all samples. Sample TMW36042021 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 04-12-21. Samples were prepared (digested) for both total and dissolved metals on 04-20-21. Samples and QC samples were analyzed on 05-05-21and 05-06-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.10.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of each analysis day on 05-05-21 and 05-06-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method's acceptance criteria. The initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

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

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

4.10.3. 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. LCS/LCSD recoveries 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: Sample TMW16042021 was identified as field duplicate of TMW16042021D. Results for sample/sample duplicated is summarized in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW16042021 µg/L	TMW16042021D µg/L	**% RPD**	TMW16042021 µg/L	TMW16042021D µg/L	**% RPD**
Aluminum	U	190J	200	120	U	200
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	11	13	16.7	12	11	8.69
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	3900	4100	5.0	4100	3800	7.59
Chromium	U	2.2J	200	1.4J	U	200
Cobalt	U	U	--	U	U	--
Copper	U	3.4J	200	3.5J	3.0J	15.4
Iron	U	U	--	U	U	--
Lead	U	U	--	U	U	--
Magnesium	430J	460J	6.74	430J	400J	7.23
Manganese	3.1J	4.8J	43.0	4.2J	2.0J	71.0
Nickel	7.4	10	29.9	10	7.4	29.9
Potassium	470J	450J	4.35	460J	430J	6.74
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	440000	440000	<1	450000	450000	<1
Thallium	U	U	--	U	U	--
Vanadium	4.6J	4.3J	6.74	4.3J	3.9J	9.76
Zinc	U	U	--	U	U	--

4.10.5. Raw data was submitted for all samples. Sample TMW36042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.11. MERCURY by COLD VAPOR (Dissolved and total): 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 04-12-21. Samples were prepared (digested) on 04-26-21 and analyzed on 04-26-21 for Mercury. Samples were digested on 04-28-21 and analyzed on 04-28-21 for dissolved Mercury. 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 each analysis day on 04-26-21 and 04-28-21. The correlation coefficient of at least 0.999 was calculated to show the linearity of each 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. The QC samples consisted of one method blank and one set of LCS/LCSD for Mercury and dissolved Mercury. No sample was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD. Recoveries of LCS/LCSD were within acceptable range of 82-119%. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.11.4. Field duplicate sample and its associated sample: Sample TMW16042021 was identified as field duplicate of TMW16042021D. No Mercury was detected in sample and corresponding field duplicate sample for total and dissolved Mercury.

4.11.5. Raw data was submitted for all samples. Samples TMW36042021 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 #21D169 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

6.0 REFERENCES

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

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

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

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

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

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

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #21D173 Analytical Data Package

Publication Date: 06-27-21

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of eight (8) water samples were collected on 04-14-21. EMAX Laboratories received the samples on 04-15-21 and 06-16-21. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample TMW50042021(Lab ID# D173-04) was assigned as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation. Sample TMW63042021(Lab ID# D173-08) was designated to be spiked as MS/MSD on the chain of custody. Raw data for this sample together with method blank and LCS/LCSD for each method were also cross checked with the corresponding summary table results.

Stage 2b data validation examined quality assurance/quality control (QA/QC) elements such as holding time, (both extraction and analysis), extraction logs, instrument injection logs, method blank results, QC summary results and recoveries, LODs/LOQs, summaries of initial and continuing calibrations and completeness of results for the following requested EPA methods of analysis:
EPA Method 3050B/8260C: Volatile Organics by GC-MS (8 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (6 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (6 samples)
EPA Method SW8332: Nitroglycerine and PETN (6 samples)
EPA Method 8081B: Organochlorine Pesticides (7 samples)
EPA Method 8082: Polychlorinated Biphenyls; PCBs (3 samples)

EPA Method 8151A: Chlorinated herbicides (3 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (5 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (4 samples)

EPA Method 6850: Perchlorate (7 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (7 samples)

EPA Method 7470: Mercury & Dissolved Mercury (7 samples)
EPA Method SW9056A: Nitrate-N and Nitrite (7 samples)

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

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

The SDG # 21D173 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.

Eight (8) water samples were collected on 04-14-21. EMAX Laboratories received the samples on April 15, 2021 and April 16, 2021.

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 TMW50042021 (EMAX ID #D173-04) from this sample delivery group was designated for stage 3 data review. Raw data for this sample was evaluated comprehensively. Sample TMW63042021 (EMAX ID #D173-08) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with other QC samples in this report LCS/LCSD was also reviewed in detail. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#21D173 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW30042021	21D173-01	04-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
QC14042021TB3	21D173-02	04-14-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
BGMW01042021	21D173-03	04-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW50042021	21D173-04	04-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
BGMW02042021	21D173-05	04-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D173 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW51042021	D173-06	04-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW08042021	D173-07	04-14-21	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
TMW63042021	D173-08	04-14-21		VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D173 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW63042021MS	D173-08M	04-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW63042021MSD	D173-08S	04-14-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC

TABLE 2-1

Summary of Analytical Parameters
USACE Wingate, New Mexico

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L
	Perchlorate	SW6850	0.5µg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

ANALYSIS Method	**MATRIX**	**HOLDING TIME REQUIREMENT**	**DATA QUALIFIED AS "J"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	None. Holding times were met
Anions by IC	Water	Analysis 48 hours from collection	None. Holding times were met
Dissolved and Total Mercury	Water	Analysis within 6 Months	None. Holding times were met

2.2.3 Laboratory and Field Blanks

The objective of laboratory and field blanks is to determine the presence and extent of contamination resulting from laboratory or field activities. Blanks reported here included method and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in fifteen ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.4˚C and as high as 4.2˚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) =8/8X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =6/6X100=100%
Completeness (EPA Method 8330B: Explosives) =6/6X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =6/6X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =7/7X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =3/3X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =3/3X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 5/5X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =4/4X100=100%
Completeness (EPA Method 6850: Perchlorate) =7/7X100=100%
Completeness (EPA Method 7470: Dissolved and Total Mercury) =7/7X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =7/7X100=100%
Completeness (EPA Method SW9056A: Nitrate-N and Nitrite) =7/7X100=100%

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

4.0 DATA VALIDATION

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

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor
EPA **Method 9056A** for Nitrate and Nitrite 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. Eight water samples were collected on 04-14-21. Samples were analyzed on 04-21-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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

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

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 02-23-21 and at the beginning of analysis shift on 04-21-21. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 02-23-21. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 02-23-21
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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) 02-23-21
Tetrahydrofuran	0.9978

Average response factors for all target compounds were within method's recommended values, except for: Acetone (0.036) and 2-Butanone (0.062).

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 02-23-21
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

The initial calibration was verified by a second source standard on 02-24-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds except for Tetrahydrofuran (%D=27.1). Continuing calibration check standard was analyzed at the beginning and end of analysis shift on 04-21-21. Prior to opening continuing calibration, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

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

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

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

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for 2-Butanole (27.3%) in the opening daily standard and Naphthalene (20.5%) in the closing daily standard. This should not affect data quality.

4.1.5. Quality Control: The QC samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63042021 (lab ID #D173-08) was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for MS/MSD and LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list. However, one compound exceeded the minimum required recovery for MSD as indicated in the table below:

Target compound	TMW50042021MS %R	TMW50042021MSD %R	**Limit%**
Styrene	85.00%	73.0%*	78-123

*Exceeded the QC acceptance limit

Therefore, the result for parent sample would be qualified as estimated value, "UJ" for Styrene. 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 was collected for this sample delivery group.

4.1.7. Raw data was submitted for all samples. Sample TMW50042021 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 Bromoform, Bromodichloromethane and Dibromochloromethane were detected in trip blank. These compounds were not reported in any 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 04-14-21, extracted on 04-19-21 and were analyzed on 04-21-21 and 04-22-21.

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 03-18-21and 03-19-21 and at the beginning of each analysis shift on 04-21-21 and 04-22-21. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

Initial calibration curve was generated on 03-18-21 and 08-19-21. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-18-21
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 15%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-18-21
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 03-18-21
Benzoic acid	0.9985
2,4-Dinitrophenol	0.9990
4,6-Dinitro-2-Methylphenol	0.9982
Benzidine	0.9985
Dinoseb	0.9993
3,3-dimethylbenzidine	0.9987

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 03-19-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of each analysis shift on 04-21-21 and 04-22-21. Prior to each continuing calibration, 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 (04-21-21) I& II	Continuing cal. Response factors (04-22-21) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√
2,4-Dinitrophenol	≥ 0.01	√	√
4-Nitrophenol	≥ 0.01	√	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (04-21-21) I&II	Accepted Deviation from Initial calibration (04-22-21) I& II
Phenol	≤ 20	√	√
1,4-Dichlorobenzene	≤ 20	√	√
2-Nitrophenol	≤ 20	√	√
2,4-Dichlorophenol	≤ 20	√	√
Hexachlorobutadiene	≤ 20	√	√
4-Chloro-3-methylphenol	≤ 20	√	√
2,4,6-Trichlorophenol	≤ 20	√	√
Acenaphthene	≤ 20	√	√
N-Nitrosodiphenylamine	≤ 20	√	√
Pentachlorophenol	≤ 20	√	√
Fluoranthene	≤ 20	√	√
Di-n-Octylphthalate	≤ 20	√	√
Benzo(a)pyrene	≤ 20	√	√

√ denotes passing method acceptance limits

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

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (04-21-21) II	%Deviation from Initial calibration (04-22-21) II
Bis(2Chloroisopropylether)	≤ 20	22.0*	26.4
2,4-Dinitrophenol	≤ 20	26.5*	--
Di-n-Octylphthalate	≤ 20	21.0*	25.6
4-Nitroaniline	≤ 20	21.5*	--

*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: The QC samples reported consisted of one method blank and one set of LCS/LCSD and MS/MSD. Sample TMW63042021 was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits for the entire compound list in LCS/LCSD 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 for p-Terphenyl exceeding the lower acceptance limit in sample TMW50042021 as shown in the table below:

Field sample	p-Terphenyl as surrogate %Recovery	**Limit%**
TMW50042021	25%*	50-134

*Exceeded the QC acceptance limit

Since the rest of surrogates were all within acceptance limits, this exceedance should not affect the data quality.

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 TMW50042021 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. Seven (7) water samples were collected on 04-14-21, extracted on 04-16-21 and analyzed on 04-19-21 and 04-20-2, within the 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 02-17-21 and before sample analysis on 04-19-21 and 04-20-21. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 02-17-21 and 02-18-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 02-18-21 and 02-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

A total of five continuing calibration standards were analyzed at 10-injections interval on 04-19-21 and 04-20-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds from both channels A and B.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In all five continuing calibration standards, one mid-point concentration of 20-40µg/L was injected.

4.3.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63042021 was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

Surrogate recoveries were all within the method QC acceptance limits.

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

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4.3.6. Raw data was submitted for all samples. Sample TMW50042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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 04-14-21, extracted on 04-16-21, and analyzed on 04-20-21 and 04-21-21.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-10-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-10-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors were injected at single point for identification only.

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

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

4.4.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63042021 was designated to be spiked as MS/MSD. Percent recoveries (%R) for LCS/LCSD 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 and its associated sample: No field duplicate sample was collected for this sample delivery group.

4.4.6. Raw data was submitted for all samples. Sample TMW50042021 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 sample requested for this method. Three water samples were collected on 04-14-21, extracted on 04-19-21 and analyzed on 04-20-21.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 02-09-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list.

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

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 02-09-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-20-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all compounds from channels B. At least six compounds exceeded 20% limit in column A. Therefore, results for surrogate recoveries and QC were all calculated from channel B. Channel A 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 TMW63042021 was designated to be spiked as MS/MSD. All herbicides target list was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within established QC limits

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

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

4.5.6. Raw data was submitted for all samples. Sample TMW50042021 with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six (6) water samples requested for this method. Water samples were collected on 04-14-21, extracted on 04-21-21 and analyzed on 05-03-21 and 05-04-21, within holding time.

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

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 04-29-21. Confirmation (Kinetex-Biphenyl column) was calibrated on 01-20-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

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

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for both columns on 04-29-21 and 01-20-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of five continuing calibration standards (both primary and confirmation column) were analyzed on 05-03-21 and 05-04-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63042021 was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

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

4.6.6. Raw data was submitted for all samples. Samples TMW50042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation column.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six (6) water samples requested for this method. Water samples were collected on
04-14-21, extracted on 04-19-21 and analyzed on 04-21-21.

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

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 10-27-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit
(Less than 15 %.)

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

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-21-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63042021 was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

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

4.7.6. Raw data was submitted for all samples. Samples TMW50042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Five (5) water samples were collected on 04-14-21. Samples were analyzed on 04-15-21, 04-16-21 and 04-20-21 within holding time requirement.

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

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 03-16-21. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 03-16-21. 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 04-15-21, 04-16-21 and 04-20-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of two method blanks, and two sets of LCS/LCSD and MS/MSD. Sample TMW63042021 was designated to be spiked as MS/MSD. Percent

recoveries (%R) were within the established QC limits for LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank.
Surrogate recoveries were all within the method QC acceptance limits.

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

4.8.6. Raw data was submitted for all samples. Sample TMW50042021 was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Four (4) water samples were collected on 04-14-21, extracted on 04-19-21 and analyzed on 04-20-21.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-01-20. 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 for surrogates only. Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-01-20. 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 04-20-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63042021 was designated for MS/MSD. Percent recoveries (%R) of LCS/LCSD and MS/MSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

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

4.9.6. Raw data was submitted for all samples. Sample TMW50042021 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. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Seven (7) water samples were collected on 04-14-21. Samples were analyzed on 04-20-21.

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

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-19-21. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9998 (Perchlorate ion 83) and 0.9995 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

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

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-19-21. Percent recoveries were within required method limits (85-115% of the true value).

Total of seven continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-20-21, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW63042021 was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets of LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

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

4.10.6. Raw data was submitted for all samples. Sample TMW50042021 was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was

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

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

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven water samples requested for this method. Water samples were collected on 04-14-21. Samples were prepared (digested) for both total and dissolved metals on 04-21-21. Samples and QC samples were analyzed on 05-03-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-03-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method's acceptance criteria. The initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

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

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

4.11.3. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW63042021 was spiked as MS/MSD. LCS/LCSD recoveries were all within the acceptance limit of 80-120% for both total and dissolved metals. Recoveries of MS/MSD were all acceptable except for Sodium as listed in the table below. As a result, all positive results for parent sample will be qualified as estimated value "J". This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	TMW63042021 MS%	TMW63042021 MSD%	QC Limit%	TMW63042021 MS%	TMW63042021 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	√	√	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	-167*	-533*	85-117	-433*	-67*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

Method blank results for metals were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard. Sample TMW63042021 was used for serial dilution for total and dissolved metals. The serial dilution analysis (at 5-fold dilution) was within the required 10% difference of the initial analysis. The same sample was used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total and dissolved metals.

4.11.4. Field duplicate sample and its associated sample: NO field duplicate sample was collected with this sample delivery group.

4.11.5. Raw data was submitted for all samples. Sample TMW50042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.12. MERCURY by COLD VAPOR (Dissolved and total): EPA Method 7470A

4.12.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven (7) water samples requested for this method. Water samples were collected on 04-14-21. Samples were prepared (digested) and analyzed on 04-28-21 for Mercury. Samples for dissolved Mercury were digested and analyzed on 04-29-21. The samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of each analysis day on 04-28-21 and 04-29-21. The correlation coefficient of at least 0.999 was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of method blank, LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Sample TMW63042021 was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW63042021 was also used for serial dilution. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

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

4.12.5. Raw data was submitted for all samples. Sample TMW50042021 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.

4.13. Method SW9056A: Nitrate-N and Nitrite

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for a total of seven water samples requested for this method. Water samples were collected on 04-14-21, and were analyzed on 04-15-21 and 04-16-21 within the required 48-hour holding time.

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

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-04-21. Linear curve type with correlation coefficient of at least 0.999 was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of initial calibration on 03-04-21. Recoveries were all within 90-110% of initial value. Continuing Calibration standards at 10-injections interval were analyzed on 04-15-21 and

04-16-21. A total of eight continuing calibration standards were analyzed with re-analysis of some anions. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed in each 24-hour. It was within the assigned QC limit for each anion.

4.13.3. Quality Control Samples consisted of one method blank and LCS/LCSD, MS/MSD and sample/ sample duplicate analysis. Sample TMW63042021 was spiked as MS/MSD and also used for sample duplicate analysis. Recoveries of LCS/LCSD and MS/MSD were within 90-110 % of spiked values for Nitrate and Nitrite. Percent RPDs were less than 20% for LCS/LCSD, MS/MSD and sample/sample duplicate.

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

4.13.5. Raw data was submitted for all requested field samples. Sample TMW50042021 was designated to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #21D173 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 #21D199 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 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of seven (7) water samples were collected on 04-15-21. EMAX Laboratories received the samples on 04-16-21. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample QC15042021EB1(Lab ID# D199-06) was selected as stage 3 deliverable. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation. Sample TMW01042021(Lab ID# D199-07) was designated to be spiked as MS/MSD on the chain of custody. Raw data for this sample together with method blank and LCS/LCSD for each method were also cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (7 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (2 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (6 samples)
EPA Method SW8332: Nitroglycerine and PETN (6 samples)
EPA Method 8081B: Organochlorine Pesticides (2 samples)
EPA Method 8082: Polychlorinated Biphenyls; PCBs (1 sample)

EPA Method 8151A: Chlorinated herbicides (1 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (5 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (4 samples)

EPA Method 6850: Perchlorate (6 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (6 samples)

EPA Method 7470: Mercury & Dissolved Mercury (6 samples)
EPA Method SW9056A: Nitrate-N and Nitrite (6 samples)
EPA Method 8270 SIM: 1,4-Dioxane by 8270 SIM (1 sample)

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

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

The SDG # 21D199 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.

Seven (7) water samples were collected on 04-15-21. EMAX Laboratories received the samples on April 16, 2021.

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 QC15042021EB (EMAX ID #D199-06) from this sample delivery group was selected for stage 3 data review. Raw data for this sample was evaluated comprehensively. Sample TMW01042021 (EMAX ID #D199-07) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with other QC samples in this report LCS/LCSD was also reviewed in detail. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#21D199 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW10042021	21D199-01	04-15-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline TPH as DRO Perchlorate by 6850 Anions by IC
MW24042021	21D199-02	04-15-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
QC15042021TB4	21D199-03	04-15-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline
MW03042021	21D199-04	04-15-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline TPH as DRO Perchlorate by 6850 Anions by IC
MW03042021D	21D199-05	04-15-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline TPH as DRO Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D199 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
QC15042021EB1	21D199-06	04-15-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC 1,4-Dioxane by 8270 SIM
TMW01042021	21D199-07	04-15-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
TMW01042021MS	21D199-07M	04-15-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
TMW01042021MSD	21D199-07S	04-15-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC

TABLE 2-1

Summary of Analytical Parameters
USACE Wingate, New Mexico

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L
	Perchlorate	SW6850	0.5µg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

ANALYSIS Method	**MATRIX**	**HOLDING TIME REQUIREMENT**	**DATA QUALIFIED AS "J"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
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	None. Holding times were met
Dissolved and Total Mercury	Water	Analysis within 6 Months	None. Holding times were met

2.2.3 Laboratory and Field Blanks

The objective of laboratory and field blanks is to determine the presence and extent of contamination resulting from laboratory or field activities. Blanks reported here included method and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in six ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 2.3˚C and as high as 3.3˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

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

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

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) =7/7X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =2/2X100=100%
Completeness (EPA Method 8330B: Explosives) =6/6X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =6/6X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =2/2X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =1/1X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =1/1X100=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) =6/6X100=100%
Completeness (EPA Method 7470: Dissolved and Total Mercury) =6/6X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =6/6X100=100%
Completeness (EPA Method SW9056A: Nitrate-N and Nitrite) =6/6X100=100%
Completeness (EPA Method 3520B/8270: 1,4-Dioxane) =1/1X100=100%

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

4.0 DATA VALIDATION

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

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor
EPA **Method 9056A** for Nitrate and Nitrite by IC
EPA Method **3520C/8270 SIM** for 1,4-Dioxane by GC/MS

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

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

U	Indicates the analyses was analyzed for but not detected at or above Limit of Detection (LOD).

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

The following Reason codes were applied in the report:

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

4.1. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Seven water samples were collected on 04-15-21. Samples were analyzed on 04-21-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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

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

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-09-21 and at the beginning of analysis shift on 04-21-21. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-09-21. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-09-21
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 04-09-21
Vinyl acetate	0.9963

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.074) and 2-Butanone (0.021).

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 04-09-21
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

The initial calibration was verified by a second source standard on 04-12-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds except for Vinyl acetate (%D=20.5. Continuing calibration check standard was analyzed at the beginning and end of analysis shift on 04-21-21. Prior to each continuing calibration, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

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

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

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

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for Sec-Butylbenzene (20.6%) in the closing daily standard. This should not affect data quality.

4.1.5. Quality Control: The QC samples reported consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW01042021 (lab ID #D199-07) was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for MS/MSD and LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

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

4.1.6. **Field duplicate sample** and its associated sample: Sample MW03042021 was identified as field duplicate of MW03042021D. Traces of 1,2-Dichloroethane was detected in both sample and corresponding field duplicate sample (0.11Jµg/L in each sample and duplicate sample).

4.1.7. Raw data was submitted for all samples. Sample QC15042021EB1 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 Bromoform, Bromodichloromethane and Dibromochloromethane was detected in trip blank and equipment blank. 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. Two water samples were collected on 04-15-21, extracted on 04-19-21 and were analyzed on 04-21-21 and 04-22-21.

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 03-18-21 and 03-19-21 and at the beginning of each analysis shift on 04-21-21 and 04-22-21. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

Initial calibration curve was generated on 03-18-21 and 03-19-21. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-18-21
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 15%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-18-21
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 03-18-21
Benzoic acid	0.9985
2,4-Dinitrophenol	0.9990
4,6-Dinitro-2-Methylphenol	0.9982
Benzidine	0.9985
Dinoseb	0.9993
3,3-dimethylbenzidine	0.9987

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 03-19-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of each analysis shift on 04-21-21 and 04-22-21. Prior to each continuing calibration, 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 (04-21-21) I& II	Continuing cal. Response factors (04-22-21) I&II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√
2,4-Dinitrophenol	≥ 0.01	√	√
4-Nitrophenol	≥ 0.01	√	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (04-21-21) I&II	Accepted Deviation from Initial calibration (04-22-21) I& II
Phenol	≤ 20	√	√
1,4-Dichlorobenzene	≤ 20	√	√
2-Nitrophenol	≤ 20	√	√

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (04-21-21) I&II	Accepted Deviation from Initial calibration (04-22-21) I& II
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:

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (04-21-21) II	%Deviation from Initial calibration (04-22-21) II
Bis(2Chloroisopropylether)	≤ 20	22.0*	26.4*
2,4-Dinitrophenol	≤ 20	26.5*	--
Di-n-Octylphthalate	≤ 20	--	25.6*
4-Nitroaniline	≤ 20	21.2*	--

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

The results, 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 for this method.

4.2.7. Raw data was submitted for all samples. Sample QC15042021EB1 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. Two (2) water samples were collected on 04-15-21, extracted on 04-20-21 and analyzed on 04-22-21

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 02-17-21 and before sample analysis 04-22-21. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 02-17-21 and 02-18-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 02-18-21 and 02-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

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

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In all three 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 set of LCS/LCSD only. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

Surrogate recoveries were all within the method QC acceptance limits.

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

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4.3.6. Raw data was submitted for all samples. Sample QC15042021EB1 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.

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 sample requested for this method. One water sample was collected on 04-15-21, extracted on 04-20-21, and analyzed on 04-21-21 and 04-22-21.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-10-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-10-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors were injected at single point for identification only.

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

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

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) for 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 for this method.

4.4.6. Sample QC15042021EB1 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 sample requested for this method. One water sample was collected on 04-15-21, extracted on 04-19-21 and analyzed on 04-20-21.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 02-09-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list.

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

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 02-09-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-20-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all compounds in channel B. At least six compounds exceeded 20% limit in column A. Therefore, results for surrogate recoveries and QC were all calculated from channel B. Channel A was used for confirmation only.

4.5.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. 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 was collected for this method.

4.5.6. Sample QC15042021EB1 with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six (6) water samples requested for this method. Water samples were collected on 04-15-21, extracted on 04-21-21 and analyzed on 05-03-21 and 05-04-21, within holding time.

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

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 04-29-21. Confirmation (Kinetex-Biphenyl column) was calibrated on 01-20-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

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

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for both columns on 04-29-21 and 01-20-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of six continuing calibration standards (both primary and confirmation column) were analyzed on 05-03-21, 05-04-21and 05-12-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD. Sample TMW01042021 was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.6.5 Field duplicate sample and its associated sample**:** Sample MW03042021 was identified as field duplicate of MW03042021D. No explosive was detected in each sample and corresponding field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample QC15042021EB1 was selected to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation column.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six (6) water samples requested for this method. Water samples were collected on 04-15-21, extracted on 04-21-21 and analyzed on 04-22-21.

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

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 10-27-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (Less than 15 %.)

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

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-22-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD. Sample TMW01042021 was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.7.5. Field duplicate sample and its associated sample**:** Sample MW03042021 was identified as field duplicate of MW03042021D. No explosive was detected in each of sample and field duplicate sample.

4.7.6. Raw data was submitted for all samples. Samples QC15042021EB1 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Five (5) water samples were collected on 04-15-21. Samples were extracted and analyzed on 04-20-21 within holding time requirement.

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

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 03-16-21. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 03-16-21. 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. A total of three daily calibrations were carried out on 04-20-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. 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.

4.8.5. Field duplicate sample and its associated sample: Sample MW03042021 was identified as field duplicate of MW03042021D. No TPH-Gasoline was detected in each of sample and corresponding field duplicate sample.

4.8.6. Raw data was submitted for all samples. Sample QC15042021EB1 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water sample requested for this method. Four (4) water samples were collected on 04-15-21, extracted on 04-19-21 and analyzed on 04-20-21.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-01-20. 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 for surrogates only. Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-01-20. 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. A total of four daily calibration standards were carried out on 04-20-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

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

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

4.9.5. Field duplicate sample and its associated sample: Sample MW03042021 was identified as field duplicate of MW03042021D. No TPH-DRO was detected in each of sample and corresponding field duplicate sample.

4.9.6. Raw data was submitted for all samples. Sample QC15042021EB1 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Six (6) water samples were collected on 04-15-21. Samples were analyzed on 04-20-21 and 04-21-21 within holding time.

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

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-19-21. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9998 (Perchlorate ion 83) and 0.9995 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

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

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-19-21. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of seven daily standards were carried out on 04-20-21 and 04-21-21, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of two method blanks, two sets of LCS/LCSD and MS/MSD. Sample TMW01042021 was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets of LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.10.5. Field duplicate sample and its associated sample: Sample MW03042021 was identified as field duplicate of MW03042021D. Both sample and corresponding field duplicate sample were reported as non-detect for Perchlorate.

4.10.6. Raw data was submitted for all samples. Sample QC15042021EB1 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six water samples requested for this method. Water samples were collected on 04-15-21. Samples were prepared (digested) for both total and dissolved metals on 04-22-21. Samples and QC samples were analyzed on 05-10-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-10-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method’s acceptance criteria. The initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard was used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

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

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

4.11.3. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW01042021 was spiked as MS/MSD. LCS/LCSD recoveries were all within the acceptance limit of 80-120% for both total and dissolved metals. MS/MSD recoveries were all acceptable except for Calcium and Sodium as listed in the table below. As a result, all the positive results for parent sample will be qualified as estimated value "J" for these metals. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	TMW01042021 MS%	TMW01042021 MSD%	QC Limit%	TMW01042021 MS%	TMW01042021 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	33*	133*	87-118	100	67*	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

ANALYTE	Total Metals			Dissolved Metals		
	TMW01042021 MS%	TMW01042021 MSD%	**QC Limit%**	TMW01042021 MS%	TMW01042021 MSD%	**QC Limit%**
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	33*	33*	85-117	0.0*	100	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

Method blank results for metals were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard. Sample TMW01042021 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. The same sample was also used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total and dissolved metals.

4.11.4. Field duplicate sample and its associated sample: Sample MW03042021 was identified as field duplicate of MW03042021D. Results for sample/sample duplicated is summarized in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	MW03042021 µg/L	MW03042021D µg/L	**% RPD**	MW03042021 µg/L	MW03042021D µg/L	**% RPD**
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	11	11	<1	10	10	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	61000	61000	<1	61000	62000	1.63
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	MW03042021 µg/L	MW03042021D µg/L	% RPD	MW03042021 µg/L	MW03042021D µg/L	% RPD
Iron	U	270J	200	U	U	--
Lead	U	U	--	U	U	--
Magnesium	12000	12000	<1	12000	12000	<1
Manganese	33	34	2.08	28	28	<1
Nickel	U	U	--	5.9J	6.6J	11.2
Potassium	U	U	--	U	U	6.74
Selenium	29	28	3.51	28	28	<1
Silver	U	U	--	U	U	--
Sodium	970000	980000	1.02	960000	960000	<1
Thallium	U	U	--	U	U	--
Vanadium	U	U	--	U	U	--
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Sample QC15042021EB1 was selected to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.12. MERCURY by COLD VAPOR (Dissolved and total): EPA Method 7470A

4.12.1. 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 04-15-21. Samples were prepared (digested) and analyzed on 04-29-21 for both Mercury and dissolved Mercury. Water samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 04-29-21. The correlation coefficient of at least 0.999 was calculated to show the linearity of the initial calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of method blank, LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Sample TMW01042021 was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW01042021 was also used for serial dilution. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample MW03042021 was identified as field duplicate of MW03042021D. Both sample and corresponding field duplicate sample were reported as non-detect for Mercury and dissolved Mercury.

4.12.5. Raw data was submitted for all samples. Sample QC15042021EB1 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.

4.13. Method SW9056A: Nitrate-N and Nitrite

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for a total of six water samples requested for this method. Water samples were collected on 04-15-21, and were analyzed on 04-16-21 within the required 48-hour holding time.

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

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-29-21 and 03-30-21. Linear curve type with correlation coefficient of at least 0.999 was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration of each anion versus area count of each anion) was presented for each component. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of initial calibration on 03-30-21. Recoveries were all within 90-110% of initial value. Continuing Calibration standards at 10-injections interval were analyzed on 04-16-21 and 04-17-21. A total of five continuing calibration standards were analyzed with re-analysis of some anions. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed in each 24-hour. It was within the assigned QC limit for each anion.

4.13.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD, MS/MSD and sample/ sample duplicate analysis. Sample TMW01042021 was spiked as MS/MSD and also used for sample duplicate analysis. Recoveries of LCS/LCSD and MS/MSD were within 90-110 % of spiked values for Nitrate. Recoveries of Nitrite exceeded the upper QC limit as indicated in the table below.

	TMW01042021 MS%	TMW01042021 MSD%	**QC Limit%**
Nitrite	148*	149*	87-111

* Outside control limits

Since the recoveries are high biased and parent sample was reported as non-detect for Nitrite, no qualification is applied. Percent RPDs were less than 20% for LCS/LCSD, MS/MSD and sample/sample duplicate.

4.13.4. Field duplicate sample: and its associated sample: Sample MW03042021 was identified as field duplicate of sample MW03042021D. Results of sample and corresponding field duplicate sample are shown in the table below for target anions:

	MW03042021 mg/L	MW03042021 mg/L	%RPD
Nitrate-N	5.7	5.7	<1
Nitrite-N	U	U	--

4.13.5. Raw data was submitted for all requested field samples. Sample QC15042021EB1 was designated to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

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

4.14.1. Technical Holding Times

Holding time requirement was met for all samples. One water sample was collected on 04-15-21, extracted on 04-21-21 and were analyzed on 04-29-21; within holding time.

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

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

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

Qualification notations, if any, will be summarized in result section; section 4.14.7.

4.14.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-29-21 and at the beginning of analysis batch on 04-29-21. It passed all the method assigned criteria.

4.14.3. Initial Calibration

Sample and QC samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-29-21. A multi-level calibration standard ranging from 0.05mg/L to 50mg/L was used for this purpose. Internal standard curve type in Selective Ion Monitoring (SIM) mode was used for initial calibration and all following analysis.

1,4-Dioxane-d8 was used as internal standard. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table. Average response curve type was used for 1,4-Dioxane and Bromobenzene as surrogate. Target compounds met the maximum 15% RSD limit (all less than 15%).

4.14.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-29-21. Percent difference (%D) were within ±20% of the expected value.

Continuing calibration check standard was analyzed at the beginning and at the end of analysis shift on 04-29-21. Prior to opening continuing calibration standard, instrument performance tune check standard (BFB) was carried out. It passed all the method tuning criteria. Percent difference (%D) and/or drift for both opening and closing continuing calibration standards were within ±20% of the expected value for each daily standard.

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

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

Surrogate recoveries were all within method's acceptance limit.

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

4.14.7. Sample QC15042021EB1 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #21D199 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

6.0 REFERENCES

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

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

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

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

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

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

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

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #21D218 Analytical Data Package

Publication Date: 06-28-21

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 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of fifteen (15) water samples were collected on 04-16-21. EMAX Laboratories received the samples on 04-17-21. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample TMW33042021(Lab ID# D218-04) was designated as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation. Sample TMW28042021(Lab ID# D218-09) was designated to be spiked as MS/MSD on the chain of custody. Raw data for this sample together with method blank and LCS/LCSD for each method were also cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (15 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (10 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (12 samples)
EPA Method SW8332: Nitroglycerine and PETN (12 samples)
EPA Method 8081B: Organochlorine Pesticides (8 samples)
EPA Method 8082: Polychlorinated Biphenyls; PCBs (5 sample)

EPA Method 8151A: Chlorinated herbicides (5 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (7 samples)

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

EPA Method 6850: Perchlorate (11 samples)

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

EPA Method 7470: Mercury & Dissolved Mercury (14 samples)
EPA Method SW9056A: Nitrate-N and Nitrite (14 samples)
EPA Method 8270 SIM: 1,4-Dioxane by 8270 SIM (1 sample)

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

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

The SDG # 21D218 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.

Fifteen (15) water samples were collected on 04-16-21. EMAX Laboratories received the samples on April 17, 2021.

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 TMW33042021 (EMAX ID #D218-04) from this sample delivery group was selected for stage 3 data review. Raw data for this sample was evaluated comprehensively. Sample TMW28042021 (EMAX ID #D218-09) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with other QC samples in this report LCS/LCSD was also reviewed in detail. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#21D218 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
BGMW10042021	21D218-01	04-16-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW02042021	21D218-02	04-16-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW02042021D	21D218-03	04-16-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW33042021	21D218-04	04-16-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline TPH as DRO Anions by IC
MW29042021	21D218-05	04-16-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury & Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline, TPH as DRO Chlorinated Herbicides Polychlorinated biphenyls (PCBs) Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D218 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW04042021	21D218-06	04-16-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
TMW07042021	21D218-07	04-16-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS TPH Gasoline, TPH as DRO Anions by IC
TMW41042021	21D218-08	04-16-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorinated Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
TMW28042021	21D218-09	04-16-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D218 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW55042021	21D218-10	04-16-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW43042021	21D218-11	04-16-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
TMW23042021	21D218-12	04-16-21	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
QC16042021EB2	21D218-13	04-16-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls 1,4-Dioxane by 8270 SIM Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D218 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW28042021	21D218-14	04-16-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC 1,4-Dioxane by 8270 SIM
QC16042021TB5	21D218-15	04-16-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;
TMW28042021MS	21D218-09M	04-16-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Anions by IC
TMW28042021MSD	21D218-09S	04-16-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Anions by IC

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

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L
	Perchlorate	SW6850	0.5µg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

ANALYSIS Method	**MATRIX**	**HOLDING TIME REQUIREMENT**	**DATA QUALIFIED AS "J"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	None. Holding times were met
Anions by IC	Water	Analysis 48 hours from collection	None. Holding times were met
Dissolved and Total Mercury	Water	Analysis within 6 Months	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 1.4˚C and as high as 3.4˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

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

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

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) =15/15X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =10/10X100=100%
Completeness (EPA Method 8330B: Explosives) =12/12X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =12/12X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =8/8X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =5/5X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =5/5X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 7/7X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =6/6X100=100%
Completeness (EPA Method 6850: Perchlorate) =11/11X100=100%
Completeness (EPA Method 7470: Dissolved and Total Mercury) =14/14X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =14/14X100=100%
Completeness (EPA Method SW9056A: Nitrate-N and Nitrite) =14/14X100=100%
Completeness (EPA Method 3520B/8270: 1,4-Dioxane) =1/1X100=100%

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

4.0 DATA VALIDATION

This data review covers 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 **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor
EPA **Method 9056A** for Nitrate and Nitrite by IC
EPA Method **3520C/8270 SIM** for 1,4-Dioxane by GC/MS

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

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

U Indicates the analyses was analyzed for but not detected at or above Limit of Detection (LOD).

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

The following Reason codes were applied in the report:

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

4.1. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Fifteen water samples were collected on 04-16-21. Samples were analyzed on 04-20-21, 04-21-21 and 04-22-21 within method's requirement for holding time. (Water samples were preserved with hydrochloric acid).

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

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

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 02-23-21 and at the beginning of each analysis shift on 04-20-21, 04-21-21 and 04-22-21. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 02-23-21. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 02-23-21
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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) 02-23-21
Tetrahydrofuran	0.9978

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.036) and 2-Butanone (0.062).

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 02-23-21
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

The initial calibration was verified by a second source standard on 02-24-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds except for Tetrahydrofuran (%D=27.1), due to linear regression curve type. Continuing calibration check standard was analyzed at the beginning and end of analysis each shift on 04-20-21, 04-21-21 and 04-22-21. Prior to analysis of each continuing calibration, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-20-21 (I&II)	Continuing cal. Response factors 04-21-21 (I&II)	Continuing cal. Response factors 04-22-21 (I&II)
Chloromethane	≥ 0.10	√	√	√
1,1-Dichloroethane	≥ 0.20	√	√	√
Chlorobenzene	≥ 0.50	√	√	√
Bromoform	≥ 0.10	√	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√	√

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

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds listed in the table below for each analysis shift. This minor difference should not affect data quality.

VOC Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-20-21)		%Deviation from Initial calibration (04-21-21)		%Deviation from Initial calibration (04-22-21)	
		I	II	I	I	I	II
Tetrahydrofuran	≤ 20%	27.1*	20.8*	--	20.8*	--	--
Naphthalene	≤ 20%	--	--	--	20.5*	--	23.0*

4.1.5. Quality Control: The QC samples reported consisted of three method blanks, three sets of LCS/LCSD and MS/MSD. Sample TMW28042021 (lab ID #D218-09) was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for MS/MSD and LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

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

4.1.6. **Field duplicate sample** and its associated sample: Sample TMW02042021 was identified as field duplicate of TMW02042021D. No Volatile organic Compounds were detected in each of sample or associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample TMW33042021 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 Bromoform, Bromodichloromethane and Dibromochloromethane were detected in trip blank and equipment blank. 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. Ten water samples were collected on 04-16-21, extracted on 04-21-21 and were analyzed on 04-23-21.

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 03-18-21 and 03-19-21 and at the beginning of each analysis shift on 04-23-21. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

Initial calibration curve was generated on 03-18-21 and 03-19-21. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-18-21
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 15%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-18-21
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 03-18-21
Benzoic acid	0.9985
2,4-Dinitrophenol	0.9990
4,6-Dinitro-2-Methylphenol	0.9982
Benzidine	0.9985
Dinoseb	0.9993
3,3-dimethylbenzidine	0.9987

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 03-19-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-23-21. Prior to continuing calibration, 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 (04-23-21) I	Continuing cal. Response factors (04-23-21) II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√
2,4-Dinitrophenol	≥ 0.01	√	√
4-Nitrophenol	≥ 0.01	√	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	**% Deviation From Initial calibration (Acceptance Limit)**	**Accepted Deviation from Initial calibration (04-23-21) I**	**Accepted Deviation from Initial calibration (04-23-21) II**
Phenol	≤ 20	√	√
1,4-Dichlorobenzene	≤ 20	√	√
2-Nitrophenol	≤ 20	√	√
2,4-Dichlorophenol	≤ 20	√	√
Hexachlorobutadiene	≤ 20	√	√
4-Chloro-3-methylphenol	≤ 20	√	√
2,4,6-Trichlorophenol	≤ 20	√	√
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:

Analytes	**%Deviation from Initial calibration Method Criteria**	**%Deviation from Initial calibration (04-23-21) I**	**%Deviation from Initial calibration (04-23-21) II**
Bis(2-Chloroisopropyl) ether	≤ 20	--	31.2*
Di-n-Octylphthalate	≤ 20	--	25.3*

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

The results, 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 for this method.

4.2.7. Raw data was submitted for all samples. Sample TMW33042021 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 (8) water samples were collected on 04-16-21, extracted on 04-20-21 and analyzed on 04-22-21.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 02-17-21 and before sample analysis 04-22-21. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 02-17-21 and 02-18-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than

15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 02-18-21 and 02-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

A total of four continuing calibration standards were analyzed at 10-injections interval on 04-22-21 and 04-23-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds from both channels A and B.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In all four 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 set of LCS/LCSD only. 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 within established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

Surrogate recoveries were all within the method QC acceptance limits.

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

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4.3.6. Raw data was submitted for all samples. Sample QC16042021EB2 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.

4.4. 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 04-16-21, extracted on 04-20-21, and analyzed on 04-21-21 and 04-22-21.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-10-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-10-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors were injected at single point for identification only.

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

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

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) for 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 for this method.

4.4.6. Raw data was submitted for all samples. Sample QC16042021EB2 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.

4.5. 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 sample requested for this method. Five water samples were collected on 04-16-21, extracted on 04-23-21 and analyzed on 05-20-21.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 05-19-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list.

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

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 05-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-20-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all compounds in both channels.

4.5.4. 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. 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 was collected for this method.

4.5.6. Raw data was submitted for all samples. Sample QC16042021EB2 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. Traces of Dinoseb was detected in the equipment blank. This compound was also detected in two field samples.

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

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 04-16-21, extracted on 04-22-21 and analyzed on 05-05-21 and 05-06-21, within holding time.

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

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 04-29-21. Confirmation (Kinetex-Biphenyl column) was calibrated on 01-20-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

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

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for both columns on 04-29-21 and 01-20-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of six continuing calibration standards (both primary and confirmation column) were analyzed on 05-05-21, 05-06-21and 05-12-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

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

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

4.6.5 Field duplicate sample and its associated sample**:** Sample TMW02042021 was identified as field duplicate of TMW02042021D. No explosive was detected in each sample and corresponding field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample QC16042021EB2 was selected to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation column.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 04-16-21, extracted on 04-22-21 and analyzed on 04-27-21.

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

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 10-27-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (Less than 15 %.)

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

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-27-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

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

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

4.7.5. Field duplicate sample and its associated sample**:** Sample TMW02042021 was identified as field duplicate of TMW02042021D. No explosive was detected in each of sample and field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample QC16042021EB2 was selected to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Seven (7) water samples were collected on 04-16-21. Samples were extracted and analyzed on 04-20-21 within holding time requirement.

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

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 03-16-21. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10).

Retention times for further sample analyses was used for peak identification and integration range.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 03-16-21. 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. A total of four daily calibrations were carried out on 04-20-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. 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.

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

4.8.6. Raw data was submitted for all samples. Sample TMW33042021 was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water sample requested for this method. Six (6) water samples were collected on 04-16-21, extracted on 04-22-21 and analyzed on 04-23-21.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-01-20. 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 for surrogates only. Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-01-20. 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. A total of two daily calibration standards were carried out on 04-23-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

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

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

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

4.9.6. Raw data was submitted for all samples. Sample TMW33042021 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. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Eleven (11) water samples were collected on 04-16-21. Samples were analyzed on 04-22-21 and 04-23-21 within holding time.

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

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-19-21. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9998 (Perchlorate ion 83) and 0.9995 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

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

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-19-21. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of seven daily standards were carried out on 04-22-21 and 04-23-21, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of two method blanks, two sets of LCS/LCSD and MS/MSD. Sample BGMW10042021 was selected by lab to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets of LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.10.5. Field duplicate sample and its associated sample: Sample TMW02042021 was identified as field duplicate of TMW02042021D. Perchlorate result for sample and corresponding field duplicate sample are shown in the table below:

	TMW02042021 µg/L	TMW02042021D µg/L	**%RPD**
Perchlorate	5.3	5.4	1.87

4.10.6. Raw data was submitted for all samples. Sample QC16042021EB2 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for fourteen water samples requested for this method. Water samples were collected on 04-16-21. Samples were prepared (digested) for both total and dissolved metals on 04-22-21. Samples and QC samples were analyzed on 05-10-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-10-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method's acceptance criteria. The initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

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

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

4.11.3. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW28042021 was designated to be spiked as MS/MSD. LCS/LCSD recoveries were all within the acceptance limit of 80-120% for both total and dissolved metals. Recoveries of MS/MSD were all acceptable except for Calcium, Magnesium, Manganese and Sodium as listed in the table below. As a result, all the positive results for parent sample will be qualified as estimated value "J" for these metals. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	TMW28042021 MS%	TMW02842021 MSD%	**QC Limit%**	TMW28042021 MS%	TMW28042021 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	-69*	33*	87-118	67*	33*	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	33*	107	83-118	130*	83	83-118
Manganese	43*	120*	87-115	130*	83*	87-115
Nickel	√	√	85-117	√	√	85-117
Potassium	√	√	85-115	√	√	85-115
Selenium	√	√	80-120	√	√	80-120
Silver	√	√	85-116	√	√	85-116
Sodium	67*	0.0*	85-117	233*	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. Sample TMW28042021 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. The same sample was also used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total and dissolved metals.

4.11.4. Field duplicate sample and its associated sample: Sample MW02042021 was identified as field duplicate of MW02042021D. Results for sample/sample duplicate is summarized in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	MW02042021 µg/L	MW02042021D µg/L	% RPD	MW02042021 µg/L	MW023042021D µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	8.6J	7.6J	12.3	7.9J	7.4J	6.54
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	24000	24000	<1	24000	24000	<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	2700	2800	3.64	2800	2800	<1
Manganese	21	U	200	U	U	--
Nickel	U	U	--	U	U	--
Potassium	1200J	1200J	<1	1200J	1200J	<1
Selenium	76	73	4.0	76	76	<1
Silver	U	U	--	U	U	--
Sodium	1000000	1100000	9.52	1000000	1000000	<1
Thallium	U	U	--	U	U	--
Vanadium	38	38	<1	38	37	2.7
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Sample TMW33042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.12. MERCURY by COLD VAPOR (Dissolved and total): EPA Method 7470A

4.12.1. 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 04-16-21. Samples were prepared (digested) and analyzed on 05-03-21 for both Mercury and dissolved Mercury. Samples for dissolved Mercury analysis were preserved and filtered in the lab.

4.12.2. Initial and continuing calibration: The instrument calibration for this method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 05-03-21. The correlation coefficient of at least 0.999 was calculated to show the linearity of the initial calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of method blank, LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Sample TMW28042021 was designated to be spiked as MS/MSD this method. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for Mercury and dissolved Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW28042021 was also used for serial dilution in 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.

4.12.4. Field duplicate sample and its associated sample: Sample TMW02042021 was identified as field duplicate of TMW02042021D. Both sample and corresponding field duplicate sample were reported as non-detect for both Mercury and dissolved Mercury.

4.12.5. Raw data was submitted for all samples. Sample TMW33042021 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.

4.13. Method SW9056A: Nitrate-N and Nitrite

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

04-16-21, and were analyzed on 04-17-21 and 04-18-21 within the required 48-hour holding time.

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

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-29-21 and 03-30-21. Linear curve type with correlation coefficient of at least 0.999 was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration of each anion versus area count of each anion) was presented for each component. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of initial calibration on 03-30-21. Recoveries were all within 90-110% of initial value. Continuing Calibration standards at 10-injections interval were analyzed on 04-17-21 and 04-18-21. A total of seven continuing calibration standards were analyzed with re-analysis of some anions. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed in each 24-hour. It was within the assigned QC limit for each anion.

4.13.3. Quality Control Samples consisted of one method blank and LCS/LCSD, MS/MSD and sample/ sample duplicate analysis. Sample TMW28042021 was designated to be spiked as MS/MSD and also used for sample duplicate analysis. Recoveries of LCS/LCSD and MS/MSD were within 90-110 % of spiked values for Nitrate and Nitrite. Percent RPDs were less than 20% for LCS/LCSD, MS/MSD and sample/sample duplicate.

4.13.4. Field duplicate sample: and its associated sample: Sample TMW02042021 was identified as field duplicate of sample TMW02042021D. Results for sample and corresponding field duplicate sample are shown in the table below for target anions:

	TMW02042021 mg/L	TMW023042021 mg/L	%RPD
Nitrate-N	70	74	5.56
Nitrite-N	U	U	--

4.13.5. Raw data was submitted for all requested field samples. Sample TMW33042021 was designated to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

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

4.14.1. Technical Holding Times

Holding time requirement was met for this method. One water sample was collected on 04-16-21, extracted on 04-21-21 and were analyzed on 04-29-21; within holding time.

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

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

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

Qualification notations, if any, will be summarized in result section; section 4.14.7.

4.14.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-29-21 and at the beginning of analysis batch on 04-29-21. It passed all the method assigned criteria.

4.14.3. Initial Calibration

Sample and QC samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-29-21. A multi-level calibration standard ranging from 0.05mg/L to 50mg/L was used for this purpose. Internal standard curve type in Selective Ion Monitoring (SIM) mode was used for initial calibration and all following analysis.

1,4-Dioxane-d8 was used as internal standard. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table. Average response curve type was used for 1,4-Dioxane and Bromobenzene as surrogate. Target compounds met the maximum 15% RSD limit (all less than 15%).

4.14.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-29-21. Percent difference (%D) were within ±20% of the expected value.

Continuing calibration check standard was analyzed at the beginning and at the end of analysis shift on 04-29-21. Prior to opening continuing calibration standard, instrument performance tune check standard (BFB) was carried out. It passed all the method tuning criteria. Percent difference (%D) and/or drift for both opening and closing continuing calibration standards were within ±20% of the expected value for each daily standard.

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

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

Surrogate recoveries were all within method's acceptance limit.

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

4.14.7. Sample QC16042021EB2 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #21D218 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

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of seventeen (17) water samples were collected on 04-18-21 and 04-19-21. EMAX Laboratories received the samples on 04-20-21. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Samples MW33042021(Lab ID# D225-01) and TMW52042021(Lab ID# D225-02) were designated as stage 3 deliverable on the chain of custody. Raw data for these samples were compared to the reported summary tables for each method and went through comprehensive data validation. No sample was designated to be spiked as MS/MSD on the chain of custody. Results and recoveries of LCS/LCSD were 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 (17 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (11 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (12 samples)
EPA Method SW8332: Nitroglycerine and PETN (12 samples)
EPA Method 8081B: Organochlorine Pesticides (11 samples)

EPA Method 8082: Polychlorinated Biphenyls; PCBs (9 sample)

EPA Method 8151A: Chlorinated herbicides (9 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (10 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (9 samples)

EPA Method 6850: Perchlorate (14 samples)

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

EPA Method 7470: Mercury & Dissolved Mercury (16 samples)
EPA Method SW9056A: Nitrate-N and Nitrite (14 samples)
EPA Method 8270 SIM: 1,4-Dioxane by 8270 SIM (1 sample)

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

All the requested samples were analyzed for each of the components listed in the corresponding EPA Methods (QAPP; final version, Eco & Associate, Inc. April 2019). The evaluation indicated that all the analytical work was performed as requested on the chain of custody. The extraction and analytical holding times were met for each method and all the related samples. However, two samples (QC18042021TB6, and TMW27042021D) were analyzed for Volatile Organic Compounds by 8260C five days past 14 days required holding time. Qualifications are discussed in section 4.1.7. The deviations, if any, are discussed in Section 4.0 for each method.

The SDG # 21D225 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.

Seventeen (17) water samples were collected on 04-18-21 and 04-19-21. EMAX Laboratories received the samples on April 20, 2021.

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), where applicable
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

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

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. Two field samples MW33042021(EMAX ID#D225-01) and TMW52042021 (EMAX ID #D225-02) from this sample delivery group were designated for stage 3 data review. Raw data for these samples were evaluated comprehensively. No sample was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blanks and LCS/LCSD was reviewed in detail for each method. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries, when applicable, were evaluated
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#21D225 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW33042021	21D225-01	04-19-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW52042021	21D225-02	04-19-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW17042021	21D225-03	04-19-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW17042021D	21D225-04	04-19-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D225 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW53042021	21D225-05	04-19-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
MW34042021	21D225-06	04-19-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
MW34042021D	21D225-07	04-19-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW14A042021	21D225-08	04-19-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Total & dissolved Mercury Total & dissolved Metals by ICP-MS Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D225 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW36S042021	21D225-09	04-19-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
FW31042021	21D225-10	04-19-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Anions by IC
MW36D042021	21D225-11	04-19-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW57042021	21D225-12	04-19-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Total & dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline & TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls 1,4-Dioxane by 8270 SIM Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D225 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
QC19042021EB3	21D225-13	04-19-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC 1,4-Dioxane by 8270 SIM
TMW24042021	21D225-14	04-19-21	S3VM	VOCs by SW5030B/8260C, Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Total & dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW27042021	21D225-15	04-18-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850
TMW27042021D	21D225-16	04-18-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850
QC18042021TB6	21D225-17	04-18-21	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
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L
	Perchlorate	SW6850	0.5µg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

ANALYSIS Method	**MATRIX**	**HOLDING TIME REQUIREMENT**	**DATA QUALIFIED AS "J"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	Holding times were met Except two samples*
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	None. Holding times were met
Dissolved and Total Mercury	Water	Analysis within 6 Months	None. Holding times were met

*Samples TMW27042021D and QC18042021TB6 analyzed five 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-two ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.1˚C and as high as 4.7˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

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

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

Where:

RPD = Relative percent difference

R_1 = Result of the first duplicate or measured sample concentration

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

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

3.2.2 Accuracy

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

3.2.3 Completeness

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

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

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

Completeness (EPA Method 5030B/8260C: VOCs) =17/17X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =11/11X100=100%
Completeness (EPA Method 8330B: Explosives) =12/12X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =12/12X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =11/11X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =9/9X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =9/9X100=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) =14/14X100=100%
Completeness (EPA Method 7470: Dissolved and Total Mercury) =16/16X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =16/16X100=100%
Completeness (EPA Method SW9056A: Nitrate-N and Nitrite) =14/14X100=100%
Completeness (EPA Method 3520B/8270: 1,4-Dioxane) =1/1X100=100%

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

4.0 DATA VALIDATION

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

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor
EPA **Method 9056A** for Nitrate and Nitrite by IC
EPA Method **3520C/8270 SIM** for 1,4-Dioxane by GC/MS

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

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

U Indicates the analyses was analyzed for but not detected at or above Limit of Detection (LOD).

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

The following Reason codes were applied in the report:

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

4.1. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Seventeen water samples were collected on 04-18-21 and 04-19-21. Samples were analyzed on 04-22-21, 04-23-21 and 05-07-21. (Water samples were preserved with hydrochloric acid). Holding time requirement was met for most of the samples except sample TMW27042021D and QC18042021TB6. These samples were analyzed five days past 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 4.1.7.

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 02-23-21, 04-09-21 and at the beginning of each analysis shift on 04-22-21, 04-23-21 and 05-07-21. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to two sets of initial calibration using GC/MSD. Initial calibration curve was generated on 02-23-21 and 04-09-21. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 02-23-21	Average Response factor 04-09-21
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Bromoform	≥ 0.10	√	√
Chlorobenzene	≥ 0.50	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 02-23-21	Least Square Linear Regression (CCF) 04-09-21
Tetrahydrofuran	0.9978	--
Vinyl acetate	--	0.9963

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.036, 0.074) and 2-Butanone (0.062).

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 02-23-21	Response Factors %RSD 04-09-21
Vinyl chloride	-≤ 20%	√	√
1,1-Dichloroethene	-≤ 20%	√	√
Chloroform	-≤ 20%	√	√
1,2-Dichloropropane	-≤ 20%	√	√
Toluene	-≤ 20%	√	√
Ethyl benzene	-≤ 20%	√	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

The initial calibration was verified by a second source standard on 02-24-21 and 04-12-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds except for Tetrahydrofuran (%D=27.1), and Vinyl acetate (%D=20.5%). Continuing calibration check standard was analyzed at the beginning and end of each analysis shift on 04-22-21, 04-23-21 and 05-07-21. Prior to each continuing calibration, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-22-21 (I&II)	Continuing cal. Response factors 04-23-21 (I&II)	Continuing cal. Response factors 05-07-21 (I&II)
Chloromethane	≥ 0.10	√	√	√
1,1-Dichloroethane	≥ 0.20	√	√	√
Chlorobenzene	≥ 0.50	√	√	√
Bromoform	≥ 0.10	√	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√	√

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-22-21) I&II	%Deviation from Initial calibration (04-23-21) I&II	%Deviation from Initial calibration (05-07-21) I&II
Vinyl chloride	≤ 20%	√	√	√
1,1-Dichloroethene	≤ 20%	√	√	√
Chloroform	≤ 20%	√	√	√
1,2-Dichloropropane	≤ 20%	√	√	√
Toluene	≤ 20%	√	√	√
Ethyl benzene	≤ 20%	√	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds listed in the table below for each analysis shift. These minor differences should not affect data quality.

VOC Target Compounds	**%Deviation From Initial calibration (Acceptance Limit)**	**%Deviation from Initial calibration (04-22-21)**		**%Deviation from Initial calibration (04-23-21)**		**%Deviation from Initial calibration (05-07-21)**	
		I	**II**	**I**	**I**	**I**	**II**
Tetrahydrofuran	≤ 20%	--	--	--	--	33.3*	35.4*
2-Chloroethylvinyl ether	≤ 20%	42.6*	30.2*	39.2*	38.5*	21.2*	26.3*
Acetone	≤ 20%	--	21.6*	--	--	--	--
2-Butanone	≤ 20%	--	26.8*	--	--	--	--
4-Methyl-2-Pentanone	≤ 20%	--	24.8	--	--	--	--

*Outside acceptance limits

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

4.1.6. **Field duplicate sample** and its associated sample: Sample TMW17042021 was identified as field duplicate of sample TMW17042021D, sample MW34042021 was identified as field duplicate of sample MW34042021D and sample TMW27042021 was identified as field duplicate of sample TMW27042021D. No Volatile organic Compounds were detected in each of samples or associated field duplicate samples.

4.1.7. Raw data was submitted for all samples. SamplesMW33042021 and TMW52042021 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. Results calculated from the raw data agreed with all the results reported in data summary reports. Two samples (TMW27042021D and QC18042021TB6) were analyzed five days past holding time. Therefore, all the positive results will be qualified as estimated value “J” and non-detect results will be qualified as estimated value “UJ”. Traces of Bromoform, Bromodichloromethane and Dibromochloromethane were detected in trip blank and equipment blank. These compounds were not detected in any of the field samples.

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

4.2.1. Technical Holding Times

Holding time requirement was met for all samples. Eleven water samples were collected on 04-19-21, extracted on 04-26-21 and were analyzed on 04-29-21 and 04-30-21.

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 03-18-21 and 03-19-21 and at the beginning of each analysis shift on 04-29-21. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

Initial calibration curve was generated on 03-18-21 and 03-19-21. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-18-21
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 15%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-18-21
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 03-18-21
Benzoic acid	0.9985
2,4-Dinitrophenol	0.9990
4,6-Dinitro-2-Methylphenol	0.9982
Benzidine	0.9985
Dinoseb	0.9993
3,3-dimethylbenzidine	0.9987

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 03-19-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-29-21 and 04-30-21. Prior to continuing calibration, 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 (04-29-21) I	Continuing cal. Response factors (04-30-21) II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√
2,4-Dinitrophenol	≥ 0.01	√	√
4-Nitrophenol	≥ 0.01	√	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (04-29-21) I	Accepted Deviation from Initial calibration (04-30-21) II
Phenol	≤ 20	√	√
1,4-Dichlorobenzene	≤ 20	√	√
2-Nitrophenol	≤ 20	√	√
2,4-Dichlorophenol	≤ 20	√	√
Hexachlorobutadiene	≤ 20	√	√
4-Chloro-3-methylphenol	≤ 20	√	√
2,4,6-Trichlorophenol	≤ 20	√	√
Acenaphthene	≤ 20	√	√
N-Nitrosodiphenylamine	≤ 20	√	√
Pentachlorophenol	≤ 20	√	√
Fluoranthene	≤ 20	√	√
Di-n-Octylphthalate	≤ 20	√	√
Benzo(a)pyrene	≤ 20	√	√

√ denotes passing method acceptance limits

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

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (04-23-21) I	%Deviation from Initial calibration (04-23-21) II
Isophoron	≤ 20	--	27.2*

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

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

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

4.2.6. **Field duplicate sample** and its associated sample: Sample MW34042021 was identified as field duplicate of MW34042021D. No semi volatile target compound was detected in sample and associated field duplicate sample.

4.2.7. Raw data was submitted for all samples. Samples MW33042021 and TMW52042021 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. Eleven (11) water samples were collected on 04-19-21, extracted on 04-22-21 and analyzed on 04-23-21 and 04-24-21.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 02-17-21 and before sample analysis 04-23-21. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 02-17-21 and 02-18-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was also generated for Toxaphene for both channels. %RSD among the calibration factors was less than 15 for Toxaphene. A separate calibration curve was also established for Chlordane for each column. %RSD was less than 15 for Chlordane.

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

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 02-18-21 and 02-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

A total of four continuing calibration standards were analyzed at 10-injections interval on 04-23-21 and 04-24-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds from both channels A and B, except for DDT (%D=22.0) in closing daily standard in channel B. This minor difference should not affect the quality of data.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In the four 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 set of LCS/LCSD only. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Sample MW34042021 was identified as field duplicate of MW34042021D. No pesticide target compound was detected in sample and associated field duplicate sample.

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4.3.6. Raw data was submitted for all samples. Samples MW33042021 and TMW52042021 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. Nine water samples were collected on 04-19-21, extracted on 04-22-21, and analyzed on 04-23-21 and 04-26-21 within required holding time.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-10-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-10-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors were injected at single point for identification only.

A total of five continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-23-21 and 04-26-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for channels.

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

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) for 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: Sample MW34042021 was identified as field duplicate of MW34042021D. No Polychlorinated Biphenyls (PCBs) was detected in sample and associated field duplicate sample.

4.4.6. Raw data was submitted for all samples. Samples MW33042021 and TMW52042021 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 sample requested for this method. Nine water samples were collected on 04-19-21, extracted on 04-26-21 and analyzed on 05-21-21.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 05-19-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list.

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

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 05-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-21-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all compounds in both channels.

4.5.4. 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. All herbicides target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits

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

4.5.5. Field duplicate sample and its associated sample: Sample MW34042021 was identified as field duplicate of MW34042021D. No herbicide target compound was detected in each sample and associated field duplicate sample.

4.5.6. Raw data was submitted for all samples. Samples MW33042021 and TMW52042021 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. Results calculated from the raw data agreed with all the results reported in data summary reports.

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

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 04-19-21, extracted on 04-23-21 and analyzed on 05-06-21 and 05-07-21, within holding time.

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

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 04-29-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

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

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 04-29-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of four continuing calibration standards were analyzed on 05-06-21 and 05-07-2, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

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

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

4.6.5 Field duplicate sample and its associated sample**:** Sample MW34042021 was identified as field duplicate of MW34042021D. No explosive was detected in each sample and field duplicate sample.

4.6.6. Raw data was submitted for all samples. Samples MW33042021 and TMW52042021 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. Results calculated from the raw data agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation column.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 04-19-21, extracted on 04-23-21 and analyzed on 05-13-21.

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

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 10-27-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (Less than 15 %.)

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

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-13-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

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

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

4.7.5. Field duplicate sample and its associated sample**:** Sample MW34042021 was identified as field duplicate of MW34042021D. No explosive was detected in each of sample and field duplicate sample.

4.7.6. Raw data was submitted for all samples. Samples MW33042021 and TMW52042021 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 GRO (EPA Method 8015G)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Ten (10) water samples were collected on 04-19-21. Samples were extracted and analyzed on 04-22-21 within holding time requirement.

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

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 03-16-21. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 03-16-21. 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. A total of three daily calibrations were carried out on 04-22-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD 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.

4.8.5. Field duplicate sample and its associated sample: Sample MW34042021 was identified as field duplicate of MW34042021D. No TPH as Gasoline was detected in each of sample and field duplicate sample.

4.8.6. Raw data was submitted for all samples. Samples MW33042021 and TMW52042021 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. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water sample requested for this method. Nine (9) water samples were collected on 04-19-21, extracted on 04-22-21 and analyzed on 04-23-21.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-01-20. 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 for surrogates only. Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-01-20. 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. A total of four daily calibration standards were carried out on 04-23-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

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

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

4.9.5. Field duplicate sample and its associated sample: Sample MW34042021 was identified as field duplicate of MW34042021D. Results for sample and corresponding field duplicate sample are shown in the table below:

	MW34042021 mg/L	MW34042021D mg/L	%RPD
TPH ad DRO	0.11J	U	200

4.9.6. Raw data was submitted for all samples. Samples MW33042021 and TMW52042021 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. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Fourteen (14) water samples were collected on 04-18-21 and 04-19-21. Samples were analyzed on 04-22-21 and 04-23-21 within holding time.

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

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-19-21. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9998 (Perchlorate ion 83) and 0.9995 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

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

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-19-21. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of seven daily standards were carried out on 04-22-21 and 04-23-21, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

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

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

4.10.5. Field duplicate sample and its associated sample: Sample TMW17042021 was identified as field duplicate of sample TMW17042021D, sample MW34042021 was identified as field duplicate of sample MW34042021D and sample TMW27042021 was identified as field duplicate of sample TMW27042021D. No Perchlorate was detected in any of field samples and corresponding field duplicate samples.

4.10.6. Raw data was submitted for all samples. Samples MW33042021 and TMW52042021 were designated to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for sixteen water samples requested for this method. Water samples were collected on 04-18-21 and 04-19-21. Samples were prepared (digested) for both total and dissolved metals on 04-27-21. Samples and QC samples were analyzed on 05-17-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-17-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method's acceptance criteria. The initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

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

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

4.11.3. Quality Control samples consisted of one method blank and one set of LCS/LCSD for total and dissolved metals. Sample MW33042021 was spiked as MS/MSD for dissolved 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 for dissolved metals were all acceptable except for

Sodium as listed in the table below. As a result, the positive result for parent sample in dissolved metals will be qualified as estimated value "J" for this metal. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Dissolved Metals		
	MW33042021 MS%	MW33042021 MSD%	QC Limit%
Aluminum	√	√	84-117
Antimony	√	√	85-117
Arsenic	√	√	84-116
Barium	√	√	86-114
Beryllium	√	√	83-121
Cadmium	√	√	87-115
Calcium	√	√	87-118
Chromium	√	√	85-116
Cobalt	√	√	86-115
Copper	√	√	85-118
Iron	√	√	87-118
Lead	√	√	88-115
Magnesium	√	√	83-118
Manganese	√	√	87-115
Nickel	√	√	85-117
Potassium	√	√	85-115
Selenium	√	√	80-120
Silver	√	√	85-116
Sodium	-200*	-67*	85-117
Thallium	√	√	82-116
Vanadium	√	√	86-115
Zinc	√	√	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 MW33042021 was used for serial dilution for dissolved metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis. The same sample was also used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for dissolved metals.

4.11.4. Field duplicate sample and its associated sample: Sample TMW17042021 was identified as field duplicate of sample TMW17042021D, sample MW34042021 was identified as field duplicate of sample MW34042021D and sample TMW27042021 was identified as field duplicate of sample TMW27042021D. Results for each sample/sample duplicated is summarized in the tables below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW17042021 µg/L	TMW17042021D µg/L	**% RPD**	TMW17042021 µg/L	TMW17042021D µg/L	**% RPD**
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	14	13	7.41	13	13	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	3600	3600	<1	3300	3300	<1
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	U	U	--	U	U	--
Lead	U	U	--	U	U	--
Magnesium	560	560	<1	510	530	3.85
Manganese	11	11	<1	7.5	7.6	1.32
Nickel	U	U	--	U	U	--
Potassium	650J	640J	1.55	700J	680J	2.90
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	460000	460000	<1	450000	460000	2.2
Thallium	U	U	--	U	U	--
Vanadium	U	U	--	U	U	--
Zinc	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	MW34042021 µg/L	MW34042021D µg/L	**% RPD**	MW34042021 µg/L	MW347042021D µg/L	**% RPD**
Aluminum	870	830	4.71	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	1.5J	1.5J	<1	0.97J	0.96J	1.04
Barium	23	25	8.33	21	20	4.88
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	78000	76000	2.6	69000	68000	1.46
Chromium	2.0J	1.8J	10.5	U	U	--
Cobalt	0.87J	0.85J	2.3	U	U	--
Copper	2.8J	3.0J	6.9	U	5.0J	200
Iron	680J	620J	9.23	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	MW34042021 µg/L	MW34042021D µg/L	% RPD	MW34042021 µg/L	MW347042021D µg/L	% RPD
Lead	0.58J	0.27J	72.9	U	U	--
Magnesium	16000	16000	<1	15000	15000	<1
Manganese	790	1000	23.5	75	87	14.8
Nickel	1.8J	1.9J	5.4	U	U	--
Potassium	470J	480J	2.1	380J	390J	2.60
Selenium	1.9J	1.9J	<1	2.2J	2.0J	9.52
Silver	U	U	--	U	U	--
Sodium	1600000	1600000	<1	1600000	1600000	<1
Thallium	U	U	--	U	U	--
Vanadium	7.2	6.9	4.26	4.2J	3.7J	12.6
Zinc	U	U	--	U	U	--

ANALYTE	Total Metals			Dissolved Metals		
	TMW27042021 µg/L	TMW27042021D µg/L	% RPD	TMW27042021 µg/L	TMW27042021D µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	21	21	<1	21	21	<1
Barium	110	120	8.69	120	120	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	24000	24000	<1	24000	24000	<1
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	570J	580J	1.74	540J	540J	<1
Lead	U	U	--	U	U	--
Magnesium	6100	6100	<1	6200	6100	1.63
Manganese	560	560	<1	560	560	<1
Nickel	U	U	--	U	U	--
Potassium	420J	420J	<1	430J	450J	4.55
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	340000	340000	<1	340000	340000	<1
Thallium	U	U	--	U	U	--
Vanadium	U	U	<1	U	U	--
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Samples MW33042021 and TMW52042021 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. MERCURY by COLD VAPOR (Dissolved and total): EPA Method 7470A

4.12.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for sixteen (16) water samples requested for this method. Water samples were collected on 04-18-21 and 04-19-21. Samples were prepared (digested) on 05-05-21 and analyzed on 05-06-21 for Mercury. Samples were prepared and analyzed for dissolved Mercury on 05-10-21 Samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of each analysis day on 05-06-21 and 05-10-21. The correlation coefficient of at least 0.999 was calculated to show the linearity of each initial calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of method blank, LCS/LCSD and MS/MSD for total and dissolved Mercury. Sample TMW17042021 was spiked as MS/MSD for total and dissolved Mercury. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for total and dissolved Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW17042021 was also used for serial dilution. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample TMW17042021 was identified as field duplicate of sample TMW17042021D, sample MW34042021 was identified as field duplicate of sample MW34042021D and sample TMW27042021 was identified as field duplicate of sample TMW27042021D. No Mercury was detected in any of field samples and corresponding field duplicate samples.

4.10.6. Raw data was submitted for all samples. Samples MW33042021 and TMW52042021 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.

4.13. Method SW9056A: Nitrate-N and Nitrite

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for a total of fourteen water samples requested for this method. Water samples were collected on 04-19-21, and were analyzed on 04-20-21 and 04-21-21 within the required 48-hour holding time.

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

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-29-21 and 03-30-21. Linear curve type with correlation coefficient of at least 0.999 was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration of each anion versus area count of each anion) was presented for each component. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of initial calibration on 03-30-21. Recoveries were all within 90-110% of initial value. Continuing Calibration standards at 10-injections interval were analyzed on 04-20-21 and 04-21-21. A total of five continuing calibration standards were analyzed with re-analysis of some anions. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed in each 24-hour. It was within the assigned QC limit for each anion.

4.13.3. Quality Control Samples consisted of one method blank and LCS/LCSD, MS/MSD and sample/ sample duplicate analysis. Sample MW33042021 was spiked as MS/MSD and also used for sample duplicate analysis. Recoveries of LCS/LCSD and MS/MSD were within 90-110 % of spiked values for Nitrate. Recoveries of Nitrite exceeded the upper QC limit as indicated in the table below. Therefore, result for parent sample will be qualified as estimated value “UJ”.

	MW33042021 MS%	MW33042021 MS%	**QC Limit%**
Nitrite	118*	117*	87-111

* Outside control limits

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

4.13.4. Field duplicate sample: and its associated sample: Sample TMW17042021 was identified as field duplicate of sample TMW17042021D and sample MW34042021 was identified as field duplicate of sample MW34042021D. Results for sample and corresponding field duplicate sample are shown in the table below for target anions:

	TMW17042021 mg/L	TMW17042021 mg/L	**%RPD**
Nitrate-N	U	U	--
Nitrite-N	U	U	--

	MW34042021 mg/L	MW34042021 mg/L	**%RPD**
Nitrate-N	11	11	<1
Nitrite-N	U	U	--

4.13.5. Raw data was submitted for all requested field samples. Samples MW33042021 and TMW52042021 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. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

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

4.14.1. Technical Holding Times

Holding time requirement was met for all samples. One water sample was collected on 04-19-21, extracted on 04-26-21 and were analyzed on 04-29-21; within holding time.

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

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

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

Qualification notations, if any, will be summarized in result section; section 4.14.7.

4.14.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-29-21 and at the beginning of analysis batch on 04-29-21. It passed all the method assigned criteria.

4.14.3. Initial Calibration

Sample and QC samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-29-21. A multi-level calibration standard ranging from 0.05mg/L to 50mg/L was used for this purpose. Internal standard curve type in Selective Ion Monitoring (SIM) mode was used for initial calibration and all following analysis. 1,4-Dioxane-d8 was used as internal standard. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table. Average response curve type was used for 1,4-Dioxane and Bromobenzene as surrogate. Target compounds met the maximum 15% RSD limit (all less than 15%).

4.14.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-29-21. Percent difference (%D) were within ±20% of the expected value.

Continuing calibration check standard was analyzed at the beginning and at the end of analysis shift on 04-29-21. Prior to opening continuing calibration standard, instrument performance tune check standard (BFB) was carried out. It passed all the method tuning criteria. Percent difference (%D) and/or drift for both opening and closing continuing calibration standards were within ±20% of the expected value for each daily standard.

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

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

Surrogate recoveries were all within method's acceptance limit.

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

4.14.7. Sample QC19042021EB2 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #21D225 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose. As stated in section 4.1.7, two water samples were analyzed five days past 14-day holding time for Volatile Organic Compounds by 8260C. Results for these samples were qualified as estimated value, "UJ".

6.0 REFERENCES

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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #21D253 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 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of seven (7) water samples were collected on 04-20-21. EMAX Laboratories received the samples on 04-21-21. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample TMW45042021(Lab ID# D253-01) was designated as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation. Sample TMW34042021(Lab ID# D253-05) was designated to be spiked as MS/MSD on the chain of custody. Raw data for this sample together with method blank and LCS/LCSD for each method were also cross checked with the corresponding summary table results.

Stage 2b data validation examined quality assurance/quality control (QA/QC) elements such as holding time, (both extraction and analysis), extraction logs, instrument injection logs, method blank results, QC summary results and recoveries, LODs/LOQs, summaries of initial and continuing calibrations and completeness of results for the following requested EPA methods of analysis:
EPA Method 3050B/8260C: Volatile Organics by GC-MS (7 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (5 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (5 samples)
EPA Method SW8332: Nitroglycerine and PETN (5 samples)
EPA Method 8081B: Organochlorine Pesticides (5 samples)
EPA Method 8082: Polychlorinated Biphenyls; PCBs (3 sample)

EPA Method 8151A: Chlorinated herbicides (3 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (5 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (4 samples)

EPA Method 6850: Perchlorate (6 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (6 samples)

EPA Method 7470: Mercury & Dissolved Mercury (6 samples)
EPA Method SW9056A: Nitrate-N and Nitrite (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 # 21D253 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.

Seven (7) water samples were collected on 04-20-21. EMAX Laboratories received the samples on April 21, 2021.

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 TMW45042021 (EMAX ID #D253-01) from this sample delivery group was designated on the chain of custody for stage 3 data review. Raw data for this sample was evaluated comprehensively. Sample TMW34042021 (EMAX ID #D253-05) was designated to be spiked as MS/MSD on the chain of custody for selected methods. Raw data for this sample together with other QC samples in this report LCS/LCSD was also reviewed in detail. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#21D253 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW45042021	21D253-01	04-20-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
QC20042021TB7	21D253-02	04-20-21	S3VM	VOCs by SW5030B/8260C, TPH Gasoline
BGMW11042021	21D253-03	04-20-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
MW26042021	21D253-04	04-20-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW34042021	21D253-05	04-20-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline TPH as DRO Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D253 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation**	**Requested Methods of Analysis**
TMW64042021	21D253-06	04-20-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW31D042021	21D253-07	04-20-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-Ms Total Metals by ICP MS Perchlorate by 6850 Anions by IC
TMW34042021MS	21D253-05M	04-20-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline TPH as DRO Perchlorate by 6850 Anions by IC
TMW34042021MSD	21D253-05S	04-20-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline TPH as DRO Perchlorate by 6850 Anions by IC

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

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 μg/L
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0μg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20μg/L, (Benzidine=40μg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4μg/L
	Nitroglycerine & PETN	SW8332	120μg/L
	Chlorinated Herbicides	SW8151A	1μg/L, (MCPA=40μg/L)
	Organochlorine Pesticides	SW8081B	0.1μg/L Methoxychlor =1.0μg/L Toxaphene =2.0μg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1μg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100μg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5μg/L,1μg/L,20μg/L,100μg/L,200μg/L
	Dissolved Mercury/Mercury	SW7470A	0.5μg/L
	Anions by IC	SW9056A	0.1mg/L
	Perchlorate	SW6850	0.5μg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

ANALYSIS Method	**MATRIX**	**HOLDING TIME REQUIREMENT**	**DATA QUALIFIED AS "J"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	None. Holding times were met
Anions by IC	Water	Analysis 48 hours from collection	None. Holding times were met
Dissolved and Total Mercury	Water	Analysis within 6 Months	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 2.3˚C and as high as 5.5˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

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

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

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) =7/7X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =5/5X100=100%
Completeness (EPA Method 8330B: Explosives) =5/5X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =5/5X100=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) 5/5X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =4/4X100=100%
Completeness (EPA Method 6850: Perchlorate) =6/6X100=100%
Completeness (EPA Method 7470: Dissolved and Total Mercury) =6/6X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =6/6X100=100%
Completeness (EPA Method SW9056A: Nitrate-N and Nitrite) =6/6X100=100%

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

4.0 DATA VALIDATION

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

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor
EPA **Method 9056A** for Nitrate and Nitrite 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 04-20-21. Samples were analyzed on 04-22-21 and 04-23-21 within method’s requirement for holding time. (Water samples were preserved with hydrochloric acid).

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

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

4.1.2. Tuning criteria

The performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 02-23-21 and at the beginning of each analysis shift on 04-22-21 and 02-23-21. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 02-23-21. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 02-23-21
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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) 02-23-21**
Tetrahydrofuran	0.9978

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.036) and 2-Butanone (0.062).

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 02-23-21**
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

The initial calibration was verified by a second source standard on 02-24-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds except for Tetrahydrofuran (%D=27.1), due to linear regression curve type. Continuing calibration check standard was analyzed at the beginning and end of analysis each shift on 04-22-21 and 04-23-21. Prior to each continuing calibration, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-22-21 (I&II)	Continuing cal. Response factors 04-23-21 (I&II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-22-21) I&II	%Deviation from Initial calibration (04-23-21) I& II
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for Tetrahydrofuran and Naphthalene as listed in the table below. This minor difference should not affect data quality.

VOC Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-22-21)		%Deviation from Initial calibration (04-23-21)	
		I	II	I	II
Tetrahydrofuran	≤ 20%	31.3*	33.3*	35.4*	35.4*
Naphthalene	≤ 20%	--	23.0*	--	--

*Outside acceptance limit

4.1.5. Quality Control: The QC samples reported consisted of two method blanks, two sets of LCS/LCSD and MS/MSD. Sample TMW34042021 (lab ID #D253-05) was designated to be analyzes as MS/MSD. The full list of target compounds was spiked and reported for MS/MSD and LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blanks presented with the data package, analyzed with samples did not show presence of any target compounds.

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

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

4.1.7. Raw data was submitted for all samples. Sample TMW45042021 was designated to be reviewed as stage 3 data deliverable on the chain of custody. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports. Traces of Bromoform, Bromodichloromethane and Dibromochloromethane were detected in trip blank. 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. Five water samples were collected on 04-20-21, extracted on 04-26-21 and were analyzed on 04-29-21 and 04-30-21.

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 03-18-21 and 03-19-21 and at the beginning of analysis shift on 04-29-21. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

Initial calibration curve was generated on 03-18-21 and 03-19-21. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-18-21
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 15%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-18-21
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 03-18-21
Benzoic acid	0.9985
2,4-Dinitrophenol	0.9990
4,6-Dinitro-2-Methylphenol	0.9982
Benzidine	0.9985
Dinoseb	0.9993
3,3-dimethylbenzidine	0.9987

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 03-19-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-29-21 and 04-30-21. Prior to opening continuing calibration, 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 (04-29-21) I	Continuing cal. Response factors (04-30-21) II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√
2,4-Dinitrophenol	≥ 0.01	√	√
4-Nitrophenol	≥ 0.01	√	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (04-29-21) I	Accepted Deviation from Initial calibration (04-30-21) II
Phenol	≤ 20	√	√
1,4-Dichlorobenzene	≤ 20	√	√
2-Nitrophenol	≤ 20	√	√
2,4-Dichlorophenol	≤ 20	√	√
Hexachlorobutadiene	≤ 20	√	√
4-Chloro-3-methylphenol	≤ 20	√	√
2,4,6-Trichlorophenol	≤ 20	√	√
Acenaphthene	≤ 20	√	√
N-Nitrosodiphenylamine	≤ 20	√	√
Pentachlorophenol	≤ 20	√	√
Fluoranthene	≤ 20	√	√
Di-n-Octylphthalate	≤ 20	√	√
Benzo(a)pyrene	≤ 20	√	√

√ denotes passing method acceptance limits

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

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (04-29-21) I	%Deviation from Initial calibration (04-30-21) II
Isophorone	≤ 20	--	27.2*

*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: The QC samples reported consisted of one method blank and one set of LCS/LCSD only. No sample was assigned to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits for the entire compound list in LCS/LCSD.

The results, 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 TMW45042021 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 water samples requested for this method. Five (5) water samples were collected on 04-20-21, extracted on 04-26-21 and analyzed on 05-06-21

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 01-20-21 and before sample analysis 05-06-21. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 01-20-21 and 01-21-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was also generated for Toxaphene and Chlordane for both channels. %RSD among the calibration factors was less than 15 for Toxaphene and Chlordane.

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

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene and Chlordane, on 01-20-21 and 01-21-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

A total of three continuing calibration standards were analyzed at 10-injections interval on 05-06-21 and 05-07-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds from both channels A and B.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In the three 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 set of LCS/LCSD only. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

Surrogate recoveries were all within the method QC acceptance limits.

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

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4.3.6. Raw data was submitted for all samples. Sample TMW45042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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 04-20-21, extracted on 04-26-21, and analyzed on 04-27-21.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-10-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-10-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors were injected at single point for identification only.

A total of two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-27-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for channels.

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

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) for 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 for this sample delivery group.

4.4.6. Raw data was submitted for all samples. Sample MW26042021 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.

4.5. 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 04-20-21, extracted on 04-26-21 and analyzed on 05-21-21.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 05-19-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list.

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

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 05-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-21-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all compounds in channel both channels A and B.

4.5.4. 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. 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 was collected for this sample delivery group.

4.5.6. Raw data was submitted for all samples. Sample MW26042021 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.

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

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for five (5) water samples requested for this method. Water samples were collected on 04-20-21, extracted on 04-26-21 and analyzed on 05-12-21 and 05-13-21, within holding time.

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

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 04-29-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation

(% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

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

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte for both columns on 04-29-21. Percent difference between initial calibration response factors

(Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of two continuing calibration standards were analyzed on 05-12-21and 05-13-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

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

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

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

4.6.6. Raw data was submitted for all samples. Sample TMW45042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. All positive results, if any were confirmed with confirmation column.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for five (5) water samples requested for this method. Water samples were collected on 04-20-21, extracted on 04-26-21 and analyzed on 04-27-21.

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

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 10-27-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (Less than 15 %.)

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

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-27-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

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

Result for method blank was reviewed for each compound and no target was found in the method blank.

Surrogate recoveries were within the method QC acceptance limits.

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

4.7.6. Raw data was submitted for all samples. Sample TMW45042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Five (5) water samples were collected on 04-20-21. Samples were extracted and analyzed on 04-22-21 and 04-23-21 within holding time requirement.

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

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 03-16-21. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 03-16-21. 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. A total of four daily calibrations were carried out on 04-22-21 and 04-23-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW34042021 was designated to be 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.

4.8.5. Field duplicate sample and its associated sample: No field duplicate sample was collected with this sample data group.

4.8.6. Raw data was submitted for all samples. Sample MW26042021 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.

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

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water sample requested for this method. Four (4) water samples were collected on 04-20-21, extracted on 04-22-21 and analyzed on 04-23-21.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-01-20. 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 for surrogates only. Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-01-20. 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. A total of four daily calibration standards were carried out on 04-23-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample TMW34042021 was designated to be spiked for MS/MSD. Percent recoveries (%R) of LCS/LCSD and MS/MSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

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

4.9.6. Raw data was submitted for all samples. Sample MW26042021 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.

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

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Six (6) water samples were collected on 04-20-21. Samples were analyzed on 04-23-21 and 04-27-21 within holding time.

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

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-19-21. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^-$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9998 (Perchlorate ion 83) and 0.9995 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

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

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-19-21. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of nine daily standards were carried out on 04-23-21 and 04-27-21, bracketing the analyses of samples, sample dilutions and QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of two method blanks, two sets of LCS/LCSD and MS/MSD. Sample TMW34042021 was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets of LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

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

4.10.6. Raw data was submitted for all samples. Sample TMW45042021 was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for six water samples requested for this method. Water samples were collected on 04-20-21. Samples were prepared (digested) for both total and dissolved metals on 04-26-21. Samples and QC samples were analyzed on 05-04-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-04-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method's acceptance criteria. The initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

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

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

4.11.3. Quality Control samples consisted of method blank, one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample TMW34042021 was designated to be spiked as MS/MSD. LCS/LCSD recoveries were all within the acceptance limit of 80-120% for both total and dissolved metals. MS/MSD recoveries were all acceptable except for Calcium and Sodium as listed in the table below. As a result, all the positive results for parent sample will be qualified as estimated value "J" for these metals. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	TMW34042021 MS%	TMW34042021 MSD%	QC Limit%	TMW34042021 MS%	TMW34042021 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	0.0*	67*	87-118	100	167*	87-118
Chromium	√	√	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	√	√	85-118
Iron	√	√	87-118	√	√	87-118
Lead	√	√	88-115	√	√	88-115
Magnesium	√	√	83-118	√	√	83-118
Manganese	√	√	87-115	√	√	87-115
Nickel	√	√	85-117	√	√	85-117
Potassium	√	√	85-115	√	√	85-115
Selenium	√	√	80-120	√	√	80-120
Silver	√	√	85-116	√	√	85-116
Sodium	0.0*	-333*	85-117	2667*	-1000*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

Method blank results for metals were acceptable and no contamination was found in the method blanks, except traces of Arsenic and Selenium was detected at less than ½ Limit of quantitation (LOQ). Calibration blanks were analyzed after each continuing calibration standard. Sample TMW34042021 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. The same sample was also used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total and dissolved metals.

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

4.11.5. Raw data was submitted for all samples. Sample TMW45042021 was designated to be reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.12. MERCURY by COLD VAPOR (Dissolved and total): EPA Method 7470A

4.12.1. 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 04-20-21. Samples were prepared (digested) on 05-05-21 and analyzed on 05-06-21 for both total and dissolved Mercury. The samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for the method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 05-06-21. The correlation coefficient of at least 0.999 was calculated to show the linearity of the initial calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of method blank, LCS/LCSD and MS/MSD for total and dissolved Mercury. Sample TMW34042021 was designated to be spiked as MS/MSD for total and dissolved Mercury. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for total and dissolved Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW34042021 was also used for serial dilution. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

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

4.12.5. Raw data was submitted for all samples. Sample TMW45042021was 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.

4.13. Method SW9056A: Nitrate-N and Nitrite

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for a total of six water samples requested for this method. Water samples were collected on 04-20-21, and were analyzed on 04-21-21and 04-22-21 within the required 48-hour holding time.

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

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-29-21 and 03-30-21. Linear curve type with correlation coefficient of at least 0.999 was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of initial calibration on 03-30-21. Recoveries were all within 90-110% of initial value. Continuing Calibration standards at 10-injections interval were analyzed on 04-21-21 and 04-22-21. A total of six continuing calibration standards were analyzed with re-analysis of some anions. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed in each 24-hour. It was within the assigned QC limit for each anion.

4.13.3. Quality Control Samples consisted of one method blank, one set of LCS/LCSD, MS/MSD and sample/ sample duplicate analysis. Sample TMW34042021 was spiked as MS/MSD and also used for sample duplicate analysis. Recoveries of LCS/LCSD and MS/MSD were within 90-110 % of spiked values for Nitrate and Nitrite. Percent RPDs were less than 20% for LCS/LCSD, MS/MSD and sample/sample duplicate.

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

4.13.5. Raw data was submitted for all requested field samples. Sample TMW45042021 was designated to be reviewed as stage 3 deliverable. Raw data for this sample together with all related QC samples was reviewed for stage 3 data validation. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

5.0 CONCLUSION

SDG #21D253 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

6.0 REFERENCES

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

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

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

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

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

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

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

SDG #21D257 Analytical Data Package

Publication Date: 06-28-21

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of sixteen (16) water samples were collected on 04-21-21. EMAX Laboratories received the samples on 04-22-21. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample MW31042021(Lab ID# D257-05) was designated as stage 3 deliverable on the chain of custody. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation. 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 (16 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (10 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (12 samples)
EPA Method SW8332: Nitroglycerine and PETN (12 samples)
EPA Method 8081B: Organochlorine Pesticides (7 samples)
EPA Method 8082: Polychlorinated Biphenyls; PCBs (6 sample)
EPA Method 8151A: Chlorinated herbicides (6 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (10 samples)

EPA Method 8015D: Total Petroleum Hydrocarbons; extractable (DROs) (9 samples)

EPA Method 6850: Perchlorate (13 samples)

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

EPA Method 7470: Mercury & Dissolved Mercury (14 samples)
EPA Method SW9056A: Nitrate-N and Nitrite (15 samples)
EPA Method 8270 SIM: 1,4-Dioxane by 8270 SIM (1 sample)

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

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

The SDG # 21D257 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. However, three samples (TMW06042021, TMW29042021 and TMW35042021) were analyzed for Nitrate one to five hours past 48 hours required holding time. Qualifications are discussed in section 4.13.5. 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.

Sixteen (16) water samples were collected on 04-21-21. EMAX Laboratories received the samples on April 22, 2021.

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), where applicable
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

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

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. One field sample MW31042021 (EMAX ID #D257-05) 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 blanks and LCS/LCSD was reviewed in detail for each method. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries, when applicable, were evaluated
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#21D257 **Matrix: Water**

Field/Client ID	Lab ID	Date collected	Validation stage	Requested Methods of Analysis
MW30042021	21D257-01	04-21-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
MW18042021	21D257-02	04-21-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
MW20042021	21D257-03	04-21-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW35042021	21D257-04	04-21-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D257 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW31042021	21D257-05	04-21-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW13042021	21D257-06	04-21-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW13042021D	21D257-07	04-21-21	S3VM	VOCs by SW5030B/8260C, Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW62042021	21D257-08	04-21-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D257 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW15042021	21D257-09	04-21-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
MW25042021	21D257-10	04-21-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW06042021	21D257-11	04-21-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS TPH Gasoline; TPH as DRO Anions by IC
TMW22042021	21D257-12	04-21-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Total Mercury Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D257 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW37042021	21D257-13	04-21-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC 1,4-Dioxane by 8270 SIM
TMW29042021	21D225-14	04-21-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW35042021	21D257-15	04-21-21	S3VM	VOCs by SW5030B/8260C, Anions by IC
QC21042021TB8	21D257-16	04-18-21	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
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L
	Perchlorate	SW6850	0.5µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in nineteen 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	Holding times were met Except three samples*
Dissolved and Total Mercury	Water	Analysis within 6 Months	None. Holding times were met

*Samples TMW06042021, TMW29042021 and TMW35042021 analyzed 1-5 hours past holding time for Nitrate
Sample TMW35042021 was analyzed five hours past holding time for Nitrite.

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 nineteen ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.1˚C and as high as 5.8˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

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

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

Where:

RPD = Relative percent difference

R_1 = Result of the first duplicate or measured sample concentration

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

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

3.2.2 Accuracy

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

3.2.3 Completeness

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

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

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

Completeness (EPA Method 5030B/8260C: VOCs) =16/16X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =10/10X100=100%

Completeness (EPA Method 8330B: Explosives) =12/12X100=100%

Completeness (EPA Method 8332: Nitroglycerine & PETN) =12/12X100=100%

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

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

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

Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 10/10X100=100%

Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =9/9X100=100%
Completeness (EPA Method 6850: Perchlorate) =13/13X100=100%

Completeness (EPA Method 7470: Dissolved and Total Mercury) =14/14X100=100%

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

Completeness (EPA Method SW9056A: Nitrate-N and Nitrite) =15/15X100=100%
Completeness (EPA Method 3520B/8270: 1,4-Dioxane) =1/1X100=100%

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

4.0 DATA VALIDATION

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

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor
EPA **Method 9056A** for Nitrate and Nitrite by IC
EPA Method **3520C/8270 SIM** for 1,4-Dioxane by GC/MS

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

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

U Indicates the analyses was analyzed for but not detected at or above Limit of Detection (LOD).

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

The following Reason codes were applied in the report:

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

4.1. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Sixteen water samples were collected on 04-21-21. Samples were analyzed on 04-23-21, 04-26-21 and 04-27-21. (Water samples were preserved with hydrochloric acid). Holding time requirement was met for all samples.

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 04-15-21and at the beginning of each analysis shift on 04-23-21, 04-26-21 and 04-27-21. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-15-21. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-15-21
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 04-15-21
2-Butanole	0.9995

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.039) and 2-Butanone (0.069).

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 04-15-21
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-15-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standard was analyzed at the beginning and end of each analysis shift on 04-23-21, 04-26-21 and 04-27-21. Prior to each continuing calibration, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-23-21 (I&II)	Continuing cal. Response factors 04-26-21 (I&II)	Continuing cal. Response factors 04-27-21 (I&II)
Chloromethane	≥ 0.10	√	√	√
1,1-Dichloroethane	≥ 0.20	√	√	√
Chlorobenzene	≥ 0.50	√	√	√
Bromoform	≥ 0.10	√	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√	√

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-23-21) I&II	%Deviation from Initial calibration (04-26-21) I&II	%Deviation from Initial calibration (04-27-21) I&II
Vinyl chloride	≤ 20%	√	√	√
1,1-Dichloroethene	≤ 20%	√	√	√
Chloroform	≤ 20%	√	√	√
1,2-Dichloropropane	≤ 20%	√	√	√
Toluene	≤ 20%	√	√	√
Ethyl benzene	≤ 20%	√	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds listed in the table below for each analysis shift. These minor differences should not affect data quality.

VOC Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-23-21)		%Deviation from Initial calibration (04-26-21)		%Deviation from Initial calibration (04-27-21)	
		I	II	I	I	I	II
Trichlorofluoromethane	≤ 20%	--	34.6*	--	--	--	36.3*
2-Chloroethylvinyl ether	≤ 20%	41.7*	33.0*	41.7*	28.2*	32.0*	50.5*
Methyl acetate	≤ 20%	--	23.9*	--	--	--	24.8*

*Outside acceptance limits

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

4.1.6. **Field duplicate sample** and its associated sample: Sample TMW13042021 was identified as field duplicate of sample TMW13042021D. No Volatile Organic Compounds were detected in each of sample or associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample MW31042021 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 Bromoform, Bromodichloromethane and Dibromochloromethane were detected in trip blank. 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. Ten water samples were collected on 04-21-21, extracted on 04-27-21 and were analyzed on 04-30-21 within holding time requirement.

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 03-18-21 and 03-19-21 and at the beginning of analysis shift on 04-30-21. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

Initial calibration curve was generated on 03-18-21 and 03-19-21. A multi-level calibration standard ranging from 4mg/L to 50mg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.2.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.2.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (Method requirement)	Average Response factor 03-18-21
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 15%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-18-21
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 03-18-21
Benzoic acid	0.9985
2,4-Dinitrophenol	0.9990
4,6-Dinitro-2-Methylphenol	0.9982
Benzidine	0.9985
Dinoseb	0.9993
3,3-dimethylbenzidine	0.9987

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 03-19-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 04-30-21. Prior to continuing calibration, 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 (04-30-21) I	Continuing cal. Response factors (04-30-21) II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√
2,4-Dinitrophenol	≥ 0.01	√	√
4-Nitrophenol	≥ 0.01	√	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (04-30-21) I	Accepted Deviation from Initial calibration (04-30-21) II
Phenol	≤ 20	√	√
1,4-Dichlorobenzene	≤ 20	√	√
2-Nitrophenol	≤ 20	√	√
2,4-Dichlorophenol	≤ 20	√	√
Hexachlorobutadiene	≤ 20	√	√
4-Chloro-3-methylphenol	≤ 20	√	√
2,4,6-Trichlorophenol	≤ 20	√	√
Acenaphthene	≤ 20	√	√
N-Nitrosodiphenylamine	≤ 20	√	√
Pentachlorophenol	≤ 20	√	√
Fluoranthene	≤ 20	√	√
Di-n-Octylphthalate	≤ 20	√	√
Benzo(a)pyrene	≤ 20	√	√

√ denotes passing method acceptance limits

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

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

The results, 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 MW31042021 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. Seven (7) water samples were collected on 04-21-21, extracted on 04-26-21 and analyzed on 05-06-21 and 05-07-21.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 01-21-21 and before sample analysis 05-06-21 and 05-07-21. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 01-20-21 and 01-21-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was also generated for Toxaphene and Chlordane for both channels. %RSD among the calibration factors was less than 15 for Toxaphene and Chlordane.

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

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene and Chlordane, on 01-20-21 and 01-21-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

A total of four continuing calibration standards were analyzed at 10-injections interval on 05-06-21 and 05-07-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds from both channels A and B, except for Heptachlor (%D=23.0) in closing daily standard in channel A. This minor difference should not affect the quality of data.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. In the four 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 set of LCS/LCSD only. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

Surrogate recoveries were all within the method QC acceptance limits.

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

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4.3.6. Raw data was submitted for all samples. Sample MW31042021 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. Six water samples were collected on 04-21-21, extracted on 04-26-21, and analyzed on 04-27-21 within required holding time.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-10-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-10-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors were injected at single point for identification only.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-27-21 and 04-28-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for channels.

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

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) for 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: No field duplicate sample was assigned to this method.

4.4.6. Raw data was submitted for all samples. Sample MW31042021 was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water sample requested for this method. Six water samples were collected on 04-21-21, extracted on 04-28-21 and analyzed on 05-24-21.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 05-19-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list.

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

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 05-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-24-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all compounds in both channels except 2,4,5-T was high biased in closing daily standard (%D=30) in channel A. This compound was not detected in any of the field samples.

4.5.4. 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. 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. Samples MW31042021 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. Results calculated from the raw data agreed with all the results reported in data summary reports.

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

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 04-21-21, extracted on 04-26-21 and analyzed on 05-12-21 and 05-13-21, within holding time.

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

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 04-29-21. Confirmation (Kinetex-Biphenyl column) was calibrated on 01-20-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

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

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 04-29-21 and 01-20-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of five continuing calibration standards were analyzed on 05-12-21, 05-13-2 and 05-14-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

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

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

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

4.6.6. Raw data was submitted for all samples. Sample MW31042021 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. Results calculated from the raw data agreed with all the results reported in data summary reports. All positive results were confirmed with confirmation column.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 04-21-21, extracted on 04-26-21 and analyzed on 04-27-21.

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

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 10-27-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (Less than 15 %.)

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

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 04-27-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

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

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

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

4.7.6. Raw data was submitted for all samples. Sample MW31042021 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.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Ten (10) water samples were collected on 04-21-21. Samples were extracted and analyzed on 04-23-21and 04-24-21 within holding time requirement.

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

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 03-16-21. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 03-16-21. 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. A total of three daily calibrations were carried out on 04-23-21and 04-24-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. 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.

4.8.5. 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 MW31042021 was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water sample requested for this method. Nine (9) water samples were collected on 04-21-21, extracted on 04-27-21 and analyzed on 04-28-21, within required holding time.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-01-20. 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 for surrogates only. Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-01-20. 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. A total of two daily calibration standards were carried out on 04-28-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

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

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

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

4.9.6. Raw data was submitted for all samples. Sample MW31042021 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. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Thirteen (13) water samples were collected on 04-21-21. Samples were analyzed on 04-27-21 within holding time.

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

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-19-21. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9998 (Perchlorate ion 83) and 0.9995 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

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

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-19-21. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of six daily standards were carried out on 04-27-21 and 04-28-21, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of one method blank, one set of LCS/LCSD and MS/MSD. Sample MW35042021 was selected to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.10.5. Field duplicate sample and its associated sample: Sample TMW13042021 was identified as field duplicate of sample TMW13042021D. No Perchlorate was detected in any of field sample and corresponding field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample MW31042021 was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was

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

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

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for fourteen water samples requested for this method. Water samples were collected on 04-21-21. Samples were prepared (digested) on 04-29-21 and 04-30-21 for total and dissolved metals. Samples and QC samples were analyzed on 05-18-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-18-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method's acceptance criteria. The initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

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

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

4.11.3. Quality Control samples consisted of one method blank and one set of LCS/LCSD for total and dissolved metals. Sample MW30042021 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 for total metals failed quite a number of metals as listed in the table below. As a result, all the positive results for parent sample in total metals will be qualified as estimated value "J" for this metal. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals		
	MW30042021 MS%	MW30042021 MSD%	**QC Limit%**
Aluminum	130*	182*	84-117
Antimony	√	√	85-117
Arsenic	√	√	84-116
Barium	131*	176*	86-114
Beryllium	√	√	83-121
Cadmium	√	√	87-115
Calcium	157*	140*	87-118
Chromium	√	√	85-116
Cobalt	√	√	86-115
Copper	123*	117	85-118
Iron	125*	152*	87-118
Lead	√	√	88-115
Magnesium	108	126*	83-118
Manganese	163*	220*	87-115
Nickel	√	√	85-117
Potassium	√	√	85-115
Selenium	√	√	80-120
Silver	√	√	85-116
Sodium	1000*	633*	85-117
Thallium	√	√	82-116
Vanadium	√	√	86-115
Zinc	124*	127*	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 MW30042021 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 also used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total metals.

4.11.4. Field duplicate sample and its associated sample: Sample TMW13042021 was identified as field duplicate of sample TMW13042021D. Results for each sample/sample duplicated is summarized in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW13042021 µg/L	TMW13042021D µg/L	**% RPD**	TMW13042021 µg/L	TMW13042021D µg/L	**% RPD**
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	16	17	6.06	16	16	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	27000	27000	<1	27000	27000	<1
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	2.7J	U	200
Iron	U	U	--	U	U	--
Lead	U	U	--	U	U	--
Magnesium	5000	5100	1.98	5000	5000	<1
Manganese	U	U	--	U	U	--
Nickel	U	U	--	U	U	--
Potassium	310J	320J	3.17	360J	340J	5.71
Selenium	9.6	10	4.08	9.6	10	4.08
Silver	U	U	--	U	U	--
Sodium	560000	580000	3.51	560000	560000	<1
Thallium	U	U	--	U	U	--
Vanadium	2.1J	1.9J	10	2.0J	2.0J	<1
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Sample MW31042021 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. MERCURY by COLD VAPOR (Dissolved and total): EPA Method 7470A

4.12.1. 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

04-21-21. Samples were prepared (digested) and analyzed on 05-11-21 for Mercury. Samples were prepared on 05-10-21 and analyzed on 05-10-21 and 05-11-21 for dissolved Mercury . The samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for this method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of each analysis day on05-10-11 and 05-11-21. The correlation coefficient of at least 0.999 was calculated to show the linearity of each initial calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of method blank, LCS/LCSD and MS/MSD for total and dissolved Mercury. Sample TMW13042021 was spiked as MS/MSD for total and dissolved Mercury. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for total and dissolved Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW13042021 was also used for serial dilution. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample TMW13042021 was identified as field duplicate of sample TMW13042021D. No Mercury was detected in any of field sample and corresponding field duplicate sample.

4.12.5. Raw data was submitted for all samples. Samples MW31042021 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.

4.13. Method SW9056A: Nitrate-N and Nitrite

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for initial analysis of all water samples requested for this method. A total of fourteen (14) water samples were collected on 04-21-21, and were analyzed on 04-22-21 and 04-23-21 within the required 48-hour holding time. However, dilution analysis of three samples, TMW06042021, TMW29042021 and TMW35042021 were analyzed for Nitrate one to five hours past 48 hours required holding time. Sample TMW35042021 was also analyzed past holding time for Nitrite. Qualifications are discussed in section 4.13.5.

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

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-29-21 and 03-30-21. Linear curve type with correlation coefficient of at least 0.999 was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of initial calibration on 03-30-21. Recoveries were all within 90-110% of initial value. Continuing Calibration standards at 10-injections interval were analyzed on 04-22-21 and 04-23-21. A total of seven continuing calibration standards were analyzed with re-analysis of some anions. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed in each 24-hour. It was within the assigned QC limit for each anion.

4.13.3. Quality Control Samples consisted of one method blank and LCS/LCSD, MS/MSD and sample/ sample duplicate analysis. Sample TMW29042021 was selected to be spiked as MS/MSD and also used for sample duplicate analysis. Recoveries of LCS/LCSD and MS/MSD were within 90-110 % of spiked values for Nitrate. Recoveries of Nitrite exceeded the upper QC limit as indicated in the table below.

	TMW29042021 MS%	TMW33042021 MS%	**QC Limit%**
Nitrite	61*	61*	87-111

* Outside control limits

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

4.13.4. Field duplicate sample: and its associated sample: Sample TMW13042021 was identified as field duplicate of sample TMW13042021D. Results for sample and corresponding field duplicate sample are shown in the table below for target anions:

	TMW13042021 mg/L	TMW13042021 mg/L	**%RPD**
Nitrate-N	4.2	4.2	≤1
Nitrite-N	U	U	--

4.13.5. Raw data was submitted for all requested field samples. Samples MW31042021 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. All the samples were analyzed according to the prescribed QC procedures. Dilutions for three samples, TMW06042021, TMW29042021 and TMW35042021 were analyzed for Nitrate one to five hours past hooding time. Since the exceedance is less than two times required holding time, the positive results for these samples will be qualified as estimated value "J". Sample TMW35042021 was also analyzed past holding time for Nitrite. Therefore, result for this sample will be qualified as estimated value "UJ". All other criteria were met.

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

4.14.1. Technical Holding Times

One water sample was collected on 04-21-21, extracted on 04-26-21 and were analyzed on 04-29-21; within holding time. Selective Ion Monitoring (SIM) method with GC/MS was used for analysis.

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

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

Qualification notations, if any, will be summarized in result section; section 4.14.7.

4.14.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-29-21 and at the beginning of analysis batch on 04-29-21. It passed all the method assigned criteria.

4.14.3. Initial Calibration

Sample and QC sample were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-29-21. A multi-level calibration standard ranging from 0.05mg/L to 50mg/L was used for this purpose. Internal standard curve type in Selective Ion Monitoring (SIM) mode was used for initial calibration and all following analysis. 1,4-Dioxane-d8 was used as internal standard. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table. Average response curve type was used for 1,4-Dioxane and Bromobenzene as surrogate. Target compounds met the maximum 15% RSD limit (all less than 15%).

4.14.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-29-21. Percent difference (%D) were within ±20% of the expected value.

Continuing calibration check standard was analyzed at the beginning and at the end of analysis shift on 04-29-21. Prior to opening continuing calibration standard, instrument performance tune check standard (BFB) was carried out. It passed all the method tuning criteria. Percent difference (%D) and/or drift for both opening and closing continuing calibration standards were within ±20% of the expected value for each daily standard.

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

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

Surrogate recoveries were all within method’s acceptance limit.

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

4.14.7. Sample MW37042021 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #21D257 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose. As stated in section 4.13.5, three samples were analyzed one to five hours past 48 hour holding time for Nitrate. Positive results for these samples were qualified as estimated value, “J”.

6.0 REFERENCES

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Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

+

SDG #21D282 Analytical Data Package

Publication Date: 06-29-21

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of fifteen (15) water samples were collected on 04-23-21. EMAX Laboratories received the samples on 04-24-21. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Samples TMW47042021(Lab ID# D282-03) and TMW03042021(Lab ID# D282-04) were designated as stage 3 deliverable on the chain of custody. Raw data for these samples was compared to the reported summary tables for each method and went through comprehensive data validation. 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 (15 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (14 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (14 samples)
EPA Method SW8332: Nitroglycerine and PETN (14 samples)
EPA Method 8081B: Organochlorine Pesticides (12 samples)

EPA Method 8082: Polychlorinated Biphenyls; PCBs (7 sample)

EPA Method 8151A: Chlorinated herbicides (8 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (8 samples)

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

EPA Method 6850: Perchlorate (14 samples)

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

EPA Method 7470: Mercury & Dissolved Mercury (14 samples)
EPA Method SW9056A: Nitrate-N and Nitrite (14 samples)
EPA Method 8270 SIM: 1,4-Dioxane by 8270 SIM (3 sample)

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

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

The SDG # 21D282 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.

Fifteen (15) water samples were collected on 04-23-21. EMAX Laboratories received the samples on April 24, 2021.

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), where applicable
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

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

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. Two field samples TMW47042021 (EMAX ID #D282-03) and TMW03042021 (EMAX ID #D282-04) from this sample delivery group was designated for stage 3 data review. Raw data for these samples was evaluated comprehensively. No sample was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blanks and LCS/LCSD was reviewed in detail for each method. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries, when applicable, were evaluated
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#21D282 **Matrix: Water**

Field/Client ID	Lab ID	Date collected	Validation stage	Requested Methods of Analysis
MW27042021	21D282-01	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC 1,4-Dioxane by 8270 SIM
BGMW09042021	21D282-02	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Chlorinated Herbicides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW47042021	21D282-03	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW03042021	21D282-04	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D282 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
MW38042021	21D282-05	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC 1,4-Dioxane by 8270 SIM
MW39042021	21D282-06	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline & TPH as DRO Mercury & Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC 1,4-Dioxane by 8270 SIM
TMW48042021D	21D282-07	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
BGMW13D042021D	21D282-08	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline &TPH as DRO Mercury & Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D282 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW39D042021	21D282-09	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW49042021	21D282-10	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW40D042021	21D282-11	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
BGMW13S042021	21D282-12	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH Gasoline & TPH as DRO Mercury & Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D282 **Matrix: Water**

Field/Client ID	Lab ID	Date collected	Validation stage	Requested Methods of Analysis
BGMW13D042021	21D282-13	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
TMW59042021	21D282-14	04-23-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH Gasoline; TPH as DRO Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC
QC23042021TB10	21D282-15	04-23-21	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
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0μg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20μg/L, (Benzidine=40μg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4μg/L
	Nitroglycerine & PETN	SW8332	120μg/L
	Chlorinated Herbicides	SW8151A	1μg/L, (MCPA=40μg/L)
	Organochlorine Pesticides	SW8081B	0.1μg/L Methoxychlor =1.0μg/L Toxaphene =2.0μg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1μg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100μg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5μg/L,1μg/L,20μg/L,100μg/L,200μg/L
	Dissolved Mercury/Mercury	SW7470A	0.5μg/L
	Anions by IC	SW9056A	0.1mg/L
	Perchlorate	SW6850	0.5μg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in twenty-four 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	None. Holding times were met
Dissolved and Total Mercury	Water	Analysis within 6 Months	None. Holding times were met

2.2.3 Laboratory and Field Blanks

The objective of laboratory and field blanks is to determine the presence and extent of contamination resulting from laboratory or field activities. Blanks reported here included method and/or extraction blanks and trip blanks (VOCs and Gasoline only). The result of analysis of method blank is discussed in Section 4.0 for each method. All samples were transported in twenty-four 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 5.5˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

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

3.1 Qualitative QA Objectives

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

3.1.1 Comparability

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

3.1.2 Representativeness

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

3.2 Quantitative QA Objectives

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

3.2.1 Precision

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

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

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) =15/15X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =14/14X100=100%
Completeness (EPA Method 8330B: Explosives) =14/14X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =14/14X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =12/12X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =7/7X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =8/8X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 8/8X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =7/7X100=100%
Completeness (EPA Method 6850: Perchlorate) =14/14X100=100%
Completeness (EPA Method 7470: Dissolved and Total Mercury) =14/14X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =14/14X100=100%
Completeness (EPA Method SW9056A: Nitrate-N and Nitrite) =14/14X100=100%
Completeness (EPA Method 3520B/8270: 1,4-Dioxane) =3/3X100=100%

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

4.0 DATA VALIDATION

This data review covers 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 **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor
EPA **Method 9056A** for Nitrate and Nitrite by IC
EPA Method **3520C/8270 SIM** for 1,4-Dioxane by GC/MS

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

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

U Indicates the analyses was analyzed for but not detected at or above Limit of Detection (LOD).

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

The following Reason codes were applied in the report:

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

4.1. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Fifteen water samples were collected on 04-23-21. Samples were analyzed on 04-26-21 and 04-27-21. (Water samples were preserved with hydrochloric acid). Holding time requirement was met for all samples.

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 04-15-21and at the beginning of each analysis shift on 04-26-21 and 04-27-21. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-15-21. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-15-21
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 04-15-21
2-Butanole	0.9995

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.039) and 2-Butanone (0.069).

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 04-15-21
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-15-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standard was analyzed at the beginning and end of each analysis shift on 04-26-21 and 04-27-21. Prior to each continuing calibration, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-26-21 (I&II)	Continuing cal. Response factors 04-27-21 (I&II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-26-21) I&II	%Deviation from Initial calibration (04-27-21) I&II
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds listed in the table below for each analysis shift. These minor differences should not affect data quality.

VOC Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-26-21)		%Deviation from Initial calibration (04-27-21)	
		I	I	I	II
Trichlorofluoromethane	≤ 20%	--	--	--	36.3*
2-Chloroethylvinyl ether	≤ 20%	41.7*	28.2*	32.0*	50.5*
Methyl acetate	≤ 20%	--	--	--	24.8*

*Outside acceptance limits

4.1.5. Quality Control: The QC 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 LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blanks presented with the data package, analyzed with samples did not show presence of any target compounds.
Surrogate recoveries were all within the method’s acceptable limits.

4.1.6. **Field duplicate sample** and its associated sample: Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. No Volatile Organic Compounds were detected in each of sample or associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Samples TMW47042021 and TMW03042021were 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. Fourteen water samples were collected on 04-23-21, extracted on 04-28-21 and were analyzed on 05-03-21 within holding time requirement.

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 09-21-20 and 09-27-20 and at the beginning of analysis shift on 05-03-21. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

Initial calibration curve was generated on 09-21-20 and 09-27-20. 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 09-21-20
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 15%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 09-21-20
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) 09-21-20
Benzoic acid	0.9996
2,4-Dinitrophenol	0.9989
4,6-Dinitro-2-Methylphenol	0.9973
Di-n-octylphthalate	0.9998
Benzidine	0.9983
Dinoseb	0.9998
3,3-dimethylbenzidine	0.9987
1,3,5-Trinitrobenzene	0.9992

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 09-21-20, 09-23-20 and 10-27-20. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds, except 2,4-Dinitropheol (24.5%). Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 05-03-21. Prior to continuing calibration, 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 (05-03-21) I	Continuing cal. Response factors (05-03-21) II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√
2,4-Dinitrophenol	≥ 0.01	√	√
4-Nitrophenol	≥ 0.01	√	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (05-03-21) I	Accepted Deviation from Initial calibration (05-03-21) II
Phenol	≤ 20	√	√
1,4-Dichlorobenzene	≤ 20	√	√
2-Nitrophenol	≤ 20	√	√
2,4-Dichlorophenol	≤ 20	√	√
Hexachlorobutadiene	≤ 20	√	√
4-Chloro-3-methylphenol	≤ 20	√	√
2,4,6-Trichlorophenol	≤ 20	√	√
Acenaphthene	≤ 20	√	√
N-Nitrosodiphenylamine	≤ 20	√	√
Pentachlorophenol	≤ 20	√	√
Fluoranthene	≤ 20	√	√
Di-n-Octylphthalate	≤ 20	√	√
Benzo(a)pyrene	≤ 20	√	√

√ denotes passing method acceptance limits

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

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (05-03-21) I	%Deviation from Initial calibration (05-03-21) II
Di-n-octylphthalate	≤ 20	21.5*	24.9*
Bis(2-Chloroisopropyl) ether	≤ 20	--	22.8*

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

This minor difference should not affect the quality of presented data.

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

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

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

4.2.6. **Field duplicate sample** and its associated sample: Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. No Semi Volatile Organic Compounds were detected in each of sample or associated field duplicate sample.

4.2.7. Raw data was submitted for all samples. Samples TMW47042021 and TMW03042021were 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.

4.3. 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. Twelve (12) water samples were collected on 04-23-21, extracted on 04-29-21 and analyzed on 05-07-21, 05-08-21, 05-10-21 and 05-11-21 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.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 01-21-21 and before sample analysis 05-07-21, 05-08-21 and 05-10-21. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 01-20-21 and 01-21-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than

15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was also generated for Toxaphene and Chlordane for both channels. %RSD among the calibration factors was less than 15 for Toxaphene and Chlordane.

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

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene and Chlordane, on 01-20-21 and 01-21-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

A total of nine continuing calibration standards were analyzed at 10-injections interval on 05-07-21, 05-08-21, 05-10-21 and 05-11-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds from both channels A and B, except for few compounds in channel B.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. Therefore, this minor difference should not affect the quality of data.

In all nine 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 set of LCS/LCSD only. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. No pesticide was detected in each of sample or associated field duplicate sample.

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4.3.6. Raw data was submitted for all samples. Sample MW47042021 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. Seven water samples were collected on 04-23-21, extracted on 04-29-21, and analyzed on 05-01-21 within required holding time.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-10-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-10-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors were injected at single point for identification only.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-01-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for channels.

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

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) for 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: Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. No PCBs was detected in each of sample or associated field duplicate sample.

4.4.6. Raw data was submitted for all samples. Sample MW27042021 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 sample requested for this method. Eight water samples were collected on 04-23-21, extracted on 04-30-21 and analyzed on 05-26-21.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 05-19-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list.

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

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 05-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-26-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all compounds in both channels.

4.5.4. 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. All herbicides target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits

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

4.5.5. Field duplicate sample and its associated sample: Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. Positive herbicide results for sample and associated field duplicate sample are listed in the table below.

	BGMW13D042021 µg/L	BGMW13D042021D µg/L	**%RPD**
Dinoseb	0.29J	U	200

4.5.6. Raw data was submitted for all samples. Sample MW27042021 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. Results calculated from the raw data agreed with all the results reported in data summary reports.

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

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for fourteen (14) water samples requested for this method. Water samples were collected on 04-23-21, extracted on 04-29-21 and analyzed on 05-18-21 and 05-19-21, within holding time.

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

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on

04-29-21. Confirmation (Kinetex-Biphenyl column) was calibrated on 01-20-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

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

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 04-29-21 and 01-20-20 (confirmation column). Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of seven continuing calibration standards were analyzed on 05-18-2 and 05-19-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

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

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

4.6.5 Field duplicate sample and its associated sample**:** Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. No explosive was detected in field sample or corresponding field duplicate sample.

4.6.6. Raw data was submitted for all samples. Samples TMW47042021 and TMW03042021 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. Results calculated from the raw data agreed with all the results reported in data summary reports. All positive results were confirmed with confirmation column.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for fourteen (14) water samples requested for this method. Water samples were collected on 04-23-21, extracted on 04-29-21 and analyzed on 05-03-21.

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

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 10-27-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (Less than 15 %.)

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

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-03-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

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

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

4.7.5. Field duplicate sample and its associated sample**:** Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. No explosive was detected in field sample or corresponding field duplicate sample.

4.7.6. Raw data was submitted for all samples. Samples TMW47042021 and TMW03042021 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. Results calculated from the raw data agreed with all the results reported in data summary reports.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Eight (8) water samples were collected on 04-23-21. Samples were extracted and analyzed on 04-27-21 within holding time requirement.

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

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 03-16-21. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 03-16-21. 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. A total of three daily calibrations were carried out on 04-27-21and 04-28-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. 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.

4.8.5. Field duplicate sample and its associated sample: Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. No TPH as gasoline was detected in field sample or corresponding field duplicate sample.

4.8.6. Raw data was submitted for all samples. Sample MW27042021 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water sample requested for this method. Seven (7) water samples were collected on 04-23-21, extracted on 04-29-21 and analyzed on 04-30-21.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-01-20. 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 for surrogates only. Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-01-20. 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. A total of three daily calibration standards were carried out on 04-30-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

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

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

4.9.5. Field duplicate sample and its associated sample: Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. No TPH as DRO was detected in field sample or corresponding field duplicate sample.

4.9.6. Raw data was submitted for all samples. Sample MW27042021 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Fourteen (14) water samples were collected on 04-23-21. Samples were analyzed on 04-27-21 and 04-28-21 within holding time.

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

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-19-21. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9998 (Perchlorate ion 83) and 0.9995 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

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

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-19-21. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of nine daily standards were carried out on 04-27-21 and 04-28-21, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of two method blanks, two sets of LCS/LCSD and MS/MSD. Sample BGMW09042021 was spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits for both sets of LCS/LCSD and MS/MSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.10.5. Field duplicate sample and its associated sample: Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. No Perchlorate was detected in any of field sample and corresponding field duplicate sample.

4.10.6. Raw data was submitted for all samples. Samples MW47042021 and TMW03042021were designated to be reviewed as stage 3 data deliverable. Raw data for these samples with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for fourteen water samples requested for this method. Water samples were collected on 04-23-21. Samples were prepared (digested) on 04-30-21 for both total and dissolved metals. Samples and QC samples were analyzed on 05-19-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-19-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method's acceptance criteria. The initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

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

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

4.11.3. Quality Control samples consisted of one method blank and one set of LCS/LCSD and MS/MSD for total and dissolved metals. Sample MW27042021 was spiked 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 for total metals failed quite a number of metals as listed in the table below. As a result, all the positive results for parent sample in total metals will be qualified as estimated value "J" for this metal. This was attributed to high dilution factor and matrix interference in the case narrative.

ANALYTE	Total Metals			Dissolved Metals		
	MW27042021 MS%	MW27042021 MSD%	**QC Limit%**	MW27042021 MS%	MW27042021 MSD%	**QC Limit%**
Aluminum	117	175*	84-117	√	√	84-117
Antimony	√	√	85-117	√	√	85-117
Arsenic	√	√	84-116	√	√	84-116
Barium	122*	218*	86-114	√	√	86-114
Beryllium	√	√	83-121	√	√	83-121
Cadmium	√	√	87-115	√	√	87-115
Calcium	83*	137*	87-118	√	√	87-118
Chromium	√	√	85-116	√	√	85-116
Cobalt	√	√	86-115	√	√	86-115
Copper	√	√	85-118	√	√	85-118
Iron	114	150*	87-118	√	√	87-118
Lead	√	√	88-115	√	√	88-115
Magnesium	93	130*	83-118	√	√	83-118
Manganese	120*	173*	87-115	√	√	87-115
Nickel	√	√	85-117	86	83*	85-117
Potassium	√	√	85-115	√	√	85-115
Selenium	√	√	80-120	√	√	80-120
Silver	√	√	85-116	√	√	85-116
Sodium	-100*	-167*	85-117	367*	200*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

Method blank results for metals were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard. Sample MW27042021 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. The same sample was also used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total metals.

4.11.4. Field duplicate sample and its associated sample: Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. Results for each sample/sample duplicated is summarized in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	BGMW13D042021 µg/L	BGMW13D042021D µg/L	% RPD	BGMW13D042021 µg/L	BGMW13D042021D µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	U	--
Barium	220	230	4.44	220	220	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	15000	15000	<1	14000	14000	<1
Chromium	U	U	--	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	U	U	--
Iron	180J	180J	<1	U	U	--
Lead	U	U	--	U	U	--
Magnesium	4100	4200	2.41	4100	4000	2.41
Manganese	120	120	<1	110	110	<1
Nickel	U	U	--	U	U	--
Potassium	730J	730J	<1	730J	720J	1.38
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	270000	280000	3.64	270000	270000	<1
Thallium	U	U	--	U	U	--
Vanadium	1.9J	1.7J	11.1	1.3J	U	200
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Samples TMW47042021 and TMW03042021 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. MERCURY by COLD VAPOR (Dissolved and total): EPA Method 7470A

4.12.1. 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 04-23-21. Samples were prepared (digested) and analyzed on 05-13-21 for Mercury and dissolved Mercury. The samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for this method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 05-13-21. The correlation coefficient of at least 0.999 was calculated to show the linearity of each initial calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of method blank, LCS/LCSD and MS/MSD for total and dissolved Mercury. Sample TMW47042021 was spiked as MS/MSD for total and dissolved Mercury. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for total and dissolved Mercury. Recoveries of LCS/LCSD and MS/MSD were within acceptable range of 82-119%. Sample TMW47042021 was also used for serial dilution. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. No Mercury was detected in any of field sample and corresponding field duplicate sample.

4.12.6. Raw data was submitted for all samples. Samples TMW47042021 and TMW03042021were 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.

4.13. Method SW9056A: Nitrate-N and Nitrite

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for all water samples requested for this method. A total of fourteen (14) water samples were collected on 04-23-21, and were analyzed on 04-24-21 and 04-25-21 within the required 48-hour

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

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-29-21 and 03-30-21. A second instrument was also used and calibrated on 04-23-21. Linear curve type with correlation coefficient of at least 0.999 was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 03-30-21 and 04-23-21. Recoveries were all within 90-110% of initial value. Continuing Calibration standards at 10-injections interval were analyzed on 04-24-21 and 04-25-21. A total of nine continuing calibration standards were analyzed with re-analysis of some anions. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed in each 24-hour. It was within the assigned QC limit for each anion.

4.13.3. Quality Control Samples consisted of two method blanks, two sets of LCS/LCSD, two sets of MS/MSD and sample/ sample duplicate analysis. Samples TMW03042021 and BGMW13D042021 were spiked as MS/MSD and also used for sample duplicate analysis. Recoveries of LCS/LCSD and MS/MSD were within 90-110 % of spiked values for Nitrate. Recoveries of Nitrite exceeded the upper QC limit for each pair of MS/MSD as indicated in the table below:

	TMW03042021 MS%	TMW03042021 MSD%	**QC Limit%**
Nitrite	127*	128*	87-111
	BGMW13042021 MS%	BGMW13042021 MSD%	**QC Limit%**
Nitrite	122*	122*	87-111

* Outside control limits

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

4.13.4. Field duplicate sample: and its associated sample: Sample BGMW13D042021 was identified as field duplicate of sample BGMW13D042021D. No Nitrate and/or Nitrite was detected in any of the sample or field duplicate sample.

4.13.5. Raw data was submitted for all requested field samples. Samples MW47042021 and MW03042021 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. All the samples were analyzed according to the prescribed QC procedures. All criteria were met.

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

4.14.1. Technical Holding Times

Holding time requirement was met for all samples. Three water samples were collected on 04-23-21, extracted on 04-28-21 and were analyzed on 04-29-21; within holding time.

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

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

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

Qualification notations, if any, will be summarized in result section; section 4.14.7.

4.14.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-29-21 and at the beginning of analysis batch on 04-29-21. It passed all the method assigned criteria.

4.14.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD.

Initial calibration curve was generated on 04-29-21. A multi-level calibration standard ranging from 0.05mg/L to 50mg/L was used for this purpose. Internal standard curve type in Selective Ion Monitoring (SIM) mode was used for initial calibration and all following analysis.

1,4-Dioxane-d8 was used as internal standard. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table. Average response curve type was used for 1,4-Dioxane and Bromobenzene as surrogate. Target compounds met the maximum 15% RSD limit (all less than 15%).

4.14.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-29-21. Percent difference (%D) were within ±20% of the expected value.

Continuing calibration check standard was analyzed at the beginning and at the end of analysis shift on 04-29-21. Prior to opening continuing calibration standard, instrument performance tune check standard (BFB) was carried out. It passed all the method tuning criteria. Percent difference (%D) and/or drift for both opening and closing continuing calibration standards were within ±20% of the expected value for each daily standard.

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

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

Surrogate recoveries were all within method’s acceptance limit.

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

4.14.7. Sample MW27042021 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #21D282 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. The qualified QC data, if any, was due to matrix interference in the parent sample. Overall analytical data is of acceptable quality and considered usable for its intended purpose.

6.0 REFERENCES

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

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

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

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

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

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

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

Final Data Validation Report

USACE Fort Wingate Depot Activity
New Mexico

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

+

SDG #21D283 Analytical Data Package

Publication Date: 06-29-21

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 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of eight (8) water samples were collected on 04-22-21. EMAX Laboratories received the samples on 04-24-21. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample QC22042021EB4(Lab ID# D283-06) 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. 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 (8 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (4 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (7 samples)
EPA Method SW8332: Nitroglycerine and PETN (7 samples)
EPA Method 8081B: Organochlorine Pesticides (4 samples)
EPA Method 8082: Polychlorinated Biphenyls; PCBs (1 sample)

EPA Method 8151A: Chlorinated herbicides (1 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (4 samples)

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

EPA Method 6850: Perchlorate (5 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (7 samples)

EPA Method 7470: Mercury & Dissolved Mercury (7 samples)
EPA Method 8270 SIM: 1,4-Dioxane by 8270 SIM (1 sample)

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

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

The SDG # 21D283 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.

Eight (8) water samples were collected on 04-22-21. EMAX Laboratories received the samples on April 24, 2021.

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), where applicable
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

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

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. One field sample QC22042021EB4 (EMAX ID #D283-06) 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 blanks and LCS/LCSD was reviewed in detail for each method. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries, when applicable, were evaluated
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

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

Site Name: Fort Wingate, New Mexico

SDG#21D283 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW25042021	21D283-01	04-22-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS
TMW25042021D	21D283-02	04-22-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS
TMW38042021	21D283-03	04-22-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
QC22042021TB9	21D283-04	04-22-21	S3VM	VOCs by SW5030B/8260C, TPH as Gasoline
MW22D042021	21D283-05	04-22-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH as Gasoline TPH as DRO

Site Name: Fort Wingate, New Mexico

SDG#21D283 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
QC22042021EB4	21D283-06	04-22-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH as Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls 1,4-Dioxane by 8270 SIM
TMW32042021	21D283-07	04-22-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
MW21042021	21D283-08	04-22-21	S3VM	VOCs by SW5030B/8260C, Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH as Gasoline TPH as DRO

TABLE 2-1

Summary of Analytical Parameters
USACE Wingate, New Mexico

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L
	Perchlorate	SW6850	0.5µg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

ANALYSIS Method	**MATRIX**	**HOLDING TIME REQUIREMENT**	**DATA QUALIFIED AS "J"**
EPA Method 5030B/8260C	Water	14days to analysis (7days if **not** acid preserved)	None. Holding times were met
Semi Volatile Organic Target List 3520C/8270D/8270SIM	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroaromatics and Nitramines	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Nitroglycerine and PETN	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Chlorinated Herbicides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Organochlorine Pesticides	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Polychlorinated Biphenyls (PCBs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Total Petroleum Hydrocarbons (GROs)	Water	14days to analysis (7days if not acid preserved)	None. Holding times were met
Total Petroleum Hydrocarbons (DROs)	Water	Collection to extraction: 7 days Extraction to analysis: 40 days	None. Holding times were met
Perchlorate	Water	Collection to Analysis: 28 days	None. Holding times were met
Dissolved and Total Metals	water	Analysis within 6 Months	None. Holding times were met
Anions by IC	Water	Analysis 48 hours from collection	Not requested for this SDG
Dissolved and Total Mercury	Water	Analysis within 6 Months	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 eight ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.0˚C and as high as 4.9˚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) =8/8X100=100%

Completeness (EPA Method 3520B/8270D: SVOCs) =4/4X100=100%

Completeness (EPA Method 8330B: Explosives) =7/7X100=100%

Completeness (EPA Method 8332: Nitroglycerine & PETN) =7/7X100=100%

Completeness (EPA Method 8081B: Organochlorine pesticides) =4/4X100=100%

Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =1/1X100=100%

Completeness (EPA Method 8151B: Chlorinated Herbicides) =1/1X100=100%

Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 4/4X100=100%

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

Completeness (EPA Method 6850: Perchlorate) =5/5X100=100%

Completeness (EPA Method 7470: Dissolved and Total Mercury) =7/7X100=100%

Completeness (EPA Method 6020A: Dissolved and Total Metals) =7/7X100=100%

Completeness (EPA Method 3520B/8270: 1,4-Dioxane) =1/1X100=100%

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

4.0 DATA VALIDATION

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

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor
EPA Method **3520C/8270 SIM** for 1,4-Dioxane by GC/MS

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

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

U	Indicates the analyses was analyzed for but not detected at or above Limit of Detection (LOD).
J	Indicates an estimated value with an unknown bias.

UJ	Indicates the analyte was analyzed for but not detected and reported less than LOD. However, the numerical value is approximate.
J^+	The result was estimated value and may be biased high.
J^-	The result was estimated value and may be biased low.

X	The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality criteria. The presence or absence of the analyte cannot be confirmed by the data provided. Acceptance or rejection of the data should be decided by the project team, but exclusion of the data is recommended

The following Reason codes were applied in the report:

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

4.1. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Eight water samples were collected on 04-22-21. Samples were analyzed on 04-26-21 within required 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 04-09-21and at the beginning of analysis shift on 04-26-21. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-09-21. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-09-21
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

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

Target Analytes	Least Square Linear Regression (CCF) 04-09-21
Vinyl acetate	0.9963

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.074).

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 04-09-21
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

The initial calibration was verified by a second source standard on 04-12-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standard was analyzed at the beginning and end of analysis shift on 04-26-21. Prior to each continuing calibration, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-26-21 (I)	Continuing cal. Response factors 04-26-21 (II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-26-21) I	%Deviation from Initial calibration (04-26-21) II
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compound listed in the table below for each analysis shift. These minor differences should not affect data quality.

VOC Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-26-21)	
		I	II
2-Chloroethylvinyl ether	≤ 20%	40.5*	31.8*

*Outside acceptance limits

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

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

4.1.6. **Field duplicate sample** and its associated sample: Sample TMW25042021 was identified as field duplicate of sample TMW25042021D. No Volatile Organic Compounds were detected in each of sample or associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample QC22042021EB4 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. Traces of Bromoform, Bromodichloromethane and Dibromochloromethane were detected in trip blank. These compounds were not detected in any of the field samples. 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. Four water samples were collected on 04-22-21, extracted on 04-28-21 and were analyzed on 05-03-21 within holding time requirement.

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 09-21-20 and 09-27-20 and at the beginning of analysis shift on 05-03-21. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

Initial calibration curve was generated on 09-21-20 and 09-27-20. 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 09-21-20
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 15%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 09-21-20
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) 09-21-20
Benzoic acid	0.9996
2,4-Dinitrophenol	0.9989
4,6-Dinitro-2-Methylphenol	0.9973
Di-n-octylphthalate	0.9998
Benzidine	0.9983
Dinoseb	0.9998
3,3-dimethylbenzidine	0.9987
1,3,5-Trinitrobenzene	0.9992

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

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 09-21-20, 09-23-20 and 10-27-20. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds, except 2,4-Dinitropheol (24.5%). Continuing calibration check standards were analyzed at the beginning and at the end of analysis shift on 05-03-21. Prior to continuing calibration, 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 (05-03-21) I	Continuing cal. Response factors (05-03-21) II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√
2,4-Dinitrophenol	≥ 0.01	√	√
4-Nitrophenol	≥ 0.01	√	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (05-03-21) I	Accepted Deviation from Initial calibration (05-03-21) II
Phenol	≤ 20	√	√
1,4-Dichlorobenzene	≤ 20	√	√
2-Nitrophenol	≤ 20	√	√
2,4-Dichlorophenol	≤ 20	√	√
Hexachlorobutadiene	≤ 20	√	√
4-Chloro-3-methylphenol	≤ 20	√	√
2,4,6-Trichlorophenol	≤ 20	√	√
Acenaphthene	≤ 20	√	√
N-Nitrosodiphenylamine	≤ 20	√	√
Pentachlorophenol	≤ 20	√	√
Fluoranthene	≤ 20	√	√
Di-n-Octylphthalate	≤ 20	√	√
Benzo(a)pyrene	≤ 20	√	√

√ denotes passing method acceptance limits

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

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (05-03-21) I	%Deviation from Initial calibration (05-03-21) II
Di-n-octylphthalate	≤ 20	21.5*	24.9*
Bis(2-Chloroisopropyl) ether	≤ 20	--	22.8*

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

This minor difference should not affect the quality of presented data.

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

The results, 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 QC22042021EB4 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.

4.3. 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. Four (4) water samples were collected on 04-22-21, extracted on 04-29-21 and analyzed on 05-07-21 and 05-08-21, 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.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 01-21-21 and before sample analysis 05-07-21 and 05-08-21. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 01-20-21 and 01-21-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was also generated for Toxaphene and Chlordane for both channels. %RSD among the calibration factors was less than 15 for Toxaphene and Chlordane.

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

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene and Chlordane, on 01-20-21 and 01-21-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

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

A total of three continuing calibration standards were analyzed at 10-injections interval on 05-07-21 and 05-08-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds from both channels A and B, except for few compounds in channel B.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. Therefore, this minor difference should not affect the quality of data.

In the three 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 set of LCS/LCSD only. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

Surrogate recoveries were all within the method QC acceptance limits.

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

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4.3.6. Raw data was submitted for all samples. Sample QC22042021EB4 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.

4.4. 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 sample requested for this method. One water sample was collected on 04-22-21, extracted on 04-29-21, and analyzed on 05-01-21 within required holding time.

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

4.4.2. Initial Calibration

Initial calibration was performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-10-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%).

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

4.4.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 03-10-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors were injected at single point for identification only.

A total of two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-01-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for channels.

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

4.4.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) for 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. Sample QC22042021EB4 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.

4.5. 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 sample requested for this method. One water sample was collected on 04-22-21, extracted on 04-28-21 and analyzed on 05-24-21.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 05-19-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list.

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

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 05-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-24-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all compounds in both channels.

4.5.4. 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. 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. Sample QC22042021EB4 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.

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

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven (7) water samples requested for this method. Water samples were collected on 04-22-21, extracted on 04-28-21 and analyzed on 05-13-21 and 05-14-21, within holding time.

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

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 04-29-21. Confirmation (Kinetex-Biphenyl column) was calibrated on 01-20-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

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

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 04-29-21 and 01-20-20 (confirmation column). Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of six continuing calibration standards were analyzed on 05-13-21 and 05-14-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

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

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

4.6.5 Field duplicate sample and its associated sample**:** Sample TMW25042021 was identified as field duplicate of sample TMW25042021D. No explosive was detected in either field sample or corresponding field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample QC22042021EB4 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. All positive results were confirmed with confirmation column.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven (7) water samples requested for this method. Water samples were collected on 04-22-21, extracted on 04-28-21 and analyzed on 05-03-21.

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

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 10-27-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (Less than 15 %.)

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

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-03-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

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

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

4.7.5. Field duplicate sample and its associated sample**:** Sample TMW25042021 was identified as field duplicate of sample TMW25042021D. No explosive was detected in each of field sample or corresponding field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample QC22042021EB4 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.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Four (4) water samples were collected on 04-22-21. Samples were extracted and analyzed on 04-27-21 and 04-28-21 within holding time requirement.

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

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 03-16-21. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

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

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 03-16-21. 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. A total of four daily calibrations were carried out on 04-27-21and 04-28-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. 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.

4.8.5. 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 QC22042021EB4 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.

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

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Three (3) water samples were collected on 04-22-21, extracted on 04-29-21 and analyzed on 04-30-21.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-01-20. 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 for surrogates only. Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-01-20. 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. A total of four daily calibration standards were carried out on 04-30-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

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

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

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

4.9.6. Raw data was submitted for all samples. Sample QC22042021EB4 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.

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

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all samples requested for this method. Five (5) water samples were collected on 04-22-21. Samples were analyzed on 04-27-21 and 04-28-21 within holding time.

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

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 04-19-21. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9998 (Perchlorate ion 83) and 0.9995 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

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

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 04-19-21. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of eleven daily standards were carried out on 04-27-21 and 04-28-21, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

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

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

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

4.10.6. Raw data was submitted for all samples. Sample QC22042021EB4 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.

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

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven water samples requested for this method. Water samples were collected on 04-22-21. Samples were prepared (digested) on 04-30-21 for total and dissolved metals. Samples and QC samples were analyzed on 05-12-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-12-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method’s acceptance criteria. The initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

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

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

4.11.3. Quality Control samples consisted of one method blank and one set of LCS/LCSD for total and dissolved metals. No sample was designated to be spiked as MS/MSD for total and dissolved metals. 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.

4.11.4. Field duplicate sample and its associated sample: Sample TMW25042021 was identified as field duplicate of sample TMW25042021D. Results for each sample/sample duplicated is summarized in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW25042021 µg/L	TMW25042021D µg/L	% RPD	TMW25042021 µg/L	TMW25042021D µg/L	% RPD
Aluminum	U	U	--	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	0.64J	200	U	U	--
Barium	11	12	8.69	10	10	<1
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	50000	51000	1.98	52000	51000	1.94
Chromium	U	1.4J	200	U	U	--
Cobalt	U	U	--	U	U	--
Copper	U	U	--	4.7J	U	200
Iron	U	4400	200	U	U	--
Lead	U	U	--	U	U	--
Magnesium	10000	11000	9.53	11000	11000	<1
Manganese	160	170	6.06	81	80	1.24
Nickel	1.5J	1.9J	23.5	2.0J	1.7J	16.2
Potassium	U	U	--	U	U	--
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	870000	890000	2.27	880000	870000	1.14
Thallium	U	U	--	U	U	--
Vanadium	3.2J	3.3J	3.08	3.0J	2.9J	3.39
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Sample QC22042021EB4 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.

4.12. MERCURY by COLD VAPOR (Dissolved and total): EPA Method 7470A

4.12.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for seven (7) water samples requested for this method. Water samples were collected on 04-22-21. Samples were prepared (digested) and analyzed on 05-11-21 for Mercury and dissolved Mercury. The samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.12.2. Initial and continuing calibration: The instrument calibration for this method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of analysis day on 05-11-21. The correlation coefficient of at least 0.999 was calculated to show the linearity of each initial calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of one method blank and LCS/LCSD for each total and dissolved Mercury. No sample was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD for total and dissolved Mercury. Recoveries of LCS/LCSD were within acceptable range of 82-119%. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: Sample TMW25042021 was identified as field duplicate of sample TMW25042021D. No Mercury was detected in any of field sample and corresponding field duplicate sample.

4.12.5. Raw data was submitted for all samples. Sample QC22042021EB4 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.

4.13. 1,4-Dioxane (EPA Method 3520C/8270SIM)

4.13.1. Technical Holding Times

Holding time requirement was met for this method. One water sample was collected on 04-22-21, extracted on 04-28-21 and were analyzed on 04-29-21; within holding time.

Selective Ion Monitoring (SIM) method with GC/MS was used for analysis.

Isotopically-labeled 1,4-Dioxane-d8 was added to serve as internal standard.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.13.7.

4.13.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-29-21 and at the beginning of analysis batch on 04-29-21. It passed all the method assigned criteria.

4.13.3. Initial Calibration

Sample and QC samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-29-21. A multi-level calibration standard ranging from 0.05mg/L to 50mg/L was used for this purpose. Internal standard curve type in Selective Ion Monitoring (SIM) mode was used for initial calibration and all following analysis. 1,4-Dioxane-d8 was used as internal standard. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table. Average response curve type was used for 1,4-Dioxane and Bromobenzene as surrogate. Target compounds met the maximum 15% RSD limit (all less than 15%).

4.13.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-29-21. Percent difference (%D) were within ±20% of the expected value.

Continuing calibration check standard was analyzed at the beginning and at the end of analysis shift on 04-29-21. Prior to opening continuing calibration standard, instrument performance tune check standard (BFB) was carried out. It passed all the method tuning criteria. Percent difference (%D) and/or drift for both opening and closing continuing calibration standards were within ±20% of the expected value for each daily standard.

4.13.5. Quality Control: The QC samples reported consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. 1,4-Dioxane as the only target with Bromobenzene as surrogate was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within method's acceptance limit.

4.13.6. **Field duplicate sample** and its associated sample: No field duplicate sample was assigned for this method.

4.13.7. Sample QC22042021EB4 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #21D283 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 #21D296 Analytical Data Package

Publication Date: 06-29-2021

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EXECUTIVE SUMMARY

This data validation report presents the evaluation and validation of the analytical data for samples collected in April 2021 as part of water monitoring, Fort Wingate Depot Activity, New Mexico (NM). EMAX Laboratories in Torrance, California performed the chemical analysis of these samples. The United States Army Corps of Engineers and the State of California have certified EMAX Laboratories to perform the analysis described within this project, (QAPP, Eco & Associate, Inc. Project number Eco-18-1237, April 2019).

A total of thirteen (13) water samples were collected on 04-23-21 and 04-26-21. EMAX Laboratories received the samples on 04-27-21. The data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. Sample QC26042021EB5(Lab ID# D296-10) 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. 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 (5 samples)
EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (5 samples)
EPA Method SW8330B: Nitroaromatics and Nitramines (4 samples)
EPA Method SW8332: Nitroglycerine and PETN (4 samples)
EPA Method 8081B: Organochlorine Pesticides (6 samples)
EPA Method 8082: Polychlorinated Biphenyls; PCBs (3 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) (3 samples)

EPA Method 6850: Perchlorate (5 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (5 samples)

EPA Method 7470: Mercury & Dissolved Mercury (5 samples)
EPA Method SW9056A: Nitrate-N and Nitrite (10 samples)

EPA Method 8270 SIM: 1,4-Dioxane by 8270 SIM (1 sample)

All 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 # 21D296 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.

Thirteen (13) water samples were collected on 04-23-21 and 04-26-21. EMAX Laboratories received the samples on April 27, 2021.

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), where applicable
- Results and percent recoveries of Lab Control Samples (LCS/LCSD)

- Summary of initial calibration, initial calibration verification (ICV) and continuing calibration verification (CCV) standards;
- Case narrative for each method;
- Raw data for all the initial calibrations, initial calibration verifications, continuing calibrations, Tune check standards (where applicable), internal standard responses and chromatograms for the sample/samples at Stage 3 deliverable and related QC samples.

Data validation was performed by initial review of the analytical reports and QA/QC results and recoveries using summary tables. Next, selected analytical reports including QA/QC information was cross checked with raw data. The analysis and extraction sequence logs for each method were examined. Overall review assessed the effects of QA/QC results on the data usability. The review included such parameters as holding times, LODs/LOQs, initial and continuing calibration method requirements, surrogate recoveries, MS/MSD and lab control samples (LCS/LCSD) results and percent recoveries for accuracy and precision.

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. One field sample QC26042021EB5 (EMAX ID #D296-10) 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 blanks and LCS/LCSD was reviewed in detail for each method. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

- Analysis performed and sample identifications were verified to be in accordance with the information provided on the chain-of-custody (COC);

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries, when applicable, were evaluated
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards and instrument performance check compounds (for GC-MS) and DDT/Endrin breakdown (Method 8081B) were evaluated.

The following is a list of sample identifications and corresponding laboratory sample identification numbers:

Site Name: Fort Wingate, New Mexico

SDG#21D296 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
TMW31S042021	21D296-01	04-26-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW21042021	21D296-02	04-26-21	S3VM	Anions by IC
TMW25042021	21D296-03	04-26-21	S3VM	Anions by IC
TMW25042021D	21D296-04	04-26-21	S3VM	Anions by IC
TMW32042021	21D296-05	04-26-21	S3VM	Anions by IC
TMW38042021	21D296-06	04-26-21	S3VM	Anions by IC
MW22D042021	21D296-07	04-26-21	S3VM	Anions by IC
TMW39S042021	21D296-08	04-26-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Anions by IC
TMW61042021	21D296-09	04-26-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls TPH as Gasoline TPH as DRO Anions by IC

Site Name: Fort Wingate, New Mexico

SDG#21D296 **Matrix: Water**

Field/Client ID	**Lab ID**	**Date collected**	**Validation stage**	**Requested Methods of Analysis**
QC26042021EB5	21D296-10	04-26-21	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN TPH as Gasoline TPH as DRO Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 Chlorinated Herbicides Polychlorinated Biphenyls Anions by IC 1,4-Dioxane by 8270 SIM
QC26042021TB11	21D296-11	04-26-21	S3VM	VOCs by SW5030B/8260C, TPH as Gasoline
BGMW09042021	21D296-12	04-23-21	S3VM	Organochlorine Pesticides Polychlorinated Biphenyls
TMW35042021	21D296-13	04-26-21	S3VM	Semivolatiles + APP9 Organochlorine Pesticides Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850 TPH as DRO

TABLE 2-1
Summary of Analytical Parameters
USACE Wingate, New Mexico

Table 2-1 below shows the specified analysis for constituents in the water samples, the corresponding Environmental Protection Agency (EPA) analytical method, and the corresponding limit of quantitation (LOQ), of groups of constituents.

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	1,4-Dioxane by 8270SIM	SW3520C/8270SIM	2.0µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics &Nitramines	SW8330B	0.4µg/L
	Nitroglycerine & PETN	SW8332	120µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L,1µg/L,20µg/L,100µg/L,200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L
	Perchlorate	SW6850	0.5µg/L

2.2.1 Sample Receipt

Documentations and recordings regarding status of each sample and cooler temperatures upon receipt in the laboratory were reviewed. Samples were received in 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	Not requested for this SDG
Dissolved and Total Mercury	Water	Analysis within 6 Months	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 4.0˚C and as high as 5.8˚C upon arrival. All samples were received intact and in good condition.

3.0 QUALITY ASSURANCE OBJECTIVES

Quality assurance (QA) objectives define analytical parameters that validate the conclusions drawn from the results. Quality assurance was assessed through the following means: precision, accuracy, representativeness, completeness, and comparability (PARCC).

3.1 Qualitative QA Objectives

Qualitative aspects of QA for analytical data are characterized by completeness and representativeness.

3.1.1 Comparability

Comparability defines the level of confidence with which one data set can be compared with another. Comparability is related to accuracy and precision. It is also a measure of the data's reliability. All units for comparability are in accordance with standard procedures so that the results could be compared with other laboratories if necessary.

3.1.2 Representativeness

Representativeness is a quantity, which presents whether the results of analysis accurately portray the actual site conditions. Representativeness is a qualitative parameter, which signifies the extent of accuracy and precision, to which the data represent a characteristic population, parameter variations at a sampling point, process condition, or environmental conditions. The sampling procedures described within the approved QAPP (Eco & Associate, Inc., April 2019) are designed to provide samples representative of the site conditions.

3.2 Quantitative QA Objectives

Quantitative QA Objectives for analytical data are defined as precision, accuracy, completeness, and method quantitation limits. These quantitative parameters are established in order to monitor the overall quality of analytical data produced by the laboratory. The laboratory performing the analytical methods specified in Table 2-1, and the case narratives, which is included in the data package from the laboratory, ensures the quality of the analytical data.

3.2.1 Precision

Precision is a measure of the closeness with which multiple analyses of a given sample agree with each other. It describes the agreement between two or more measurements that have been made in exactly the same way. Precision is measured through matrix spike/matrix spike duplicate samples, laboratory control sample/ laboratory control sample duplicate and sample/sample duplicate analysis. In the latter case, the sample with positive results can be used for this purpose. The relative percent difference (RPD) is calculated as a means of quantifying precision. The following equation is used for this purpose:

$$RPD = \frac{R_1 - R_2}{(R_1 + R_2)/2} \times 100$$

Where:

RPD = Relative percent difference

R_1 = Result of the first duplicate or measured sample concentration

R_2 = Result of the second duplicate or known sample or duplicate concentration

When analytes are present at concentrations below or near the quantitation limit, precision is measured, using MS/MSD, and/or LCS/LCSD results.

Precision results are discussed in Section 4.0 of this report.

3.2.2 Accuracy

Accuracy indicates the closeness of the measurement to its true or accepted value. Accuracy measures agreement between a result and its true value. Accuracy is measured through laboratory control sample analysis and surrogate recoveries. Method-specific QA objectives for precision and accuracy were based on the quality control limits developed by the laboratory for the analytical methods, specified in Table 2-1. These procedures may affect the accuracy of the data presented. Additionally, initial and continuing calibrations were used to verify that the analytical instrument accurately measured the compound concentrations. Calculations were independently verified for the responses and percent differences (%Ds).

3.2.3 Completeness

Completeness is defined as the percentage of total measurements, which are judged to be valid. The completeness objective is to obtain a sufficient amount of valid data to enable the goals and objectives of the project to be achieved.

Completeness is quantified by computing the fraction of reports, which remained valid after the sampling procedures were reviewed and the results conformed to QA/QC protocols. The following equation was used to calculate completeness:

$$\text{Completeness} = \frac{\text{No. of valid field samples collected and analyzed}}{\text{No. of valid field samples reported}} \times 100$$

Completeness (EPA Method 5030B/8260C: VOCs) =5/5X100=100%
Completeness (EPA Method 3520B/8270D: SVOCs) =5/5X100=100%
Completeness (EPA Method 8330B: Explosives) =4/4X100=100%
Completeness (EPA Method 8332: Nitroglycerine & PETN) =4/4X100=100%
Completeness (EPA Method 8081B: Organochlorine pesticides) =6/6X100=100%
Completeness (EPA Method 8082A: Polychlorinated Biphenyls) =3/3X100=100%
Completeness (EPA Method 8151B: Chlorinated Herbicides) =2/2X100=100%
Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) 3/3X100=100%
Completeness (EPA Method 8015D: Petroleum Hydrocarbons; DRO) =3/3X100=100%
Completeness (EPA Method 6850: Perchlorate) =5/5X100=100%
Completeness (EPA Method 7470: Dissolved and Total Mercury) =5/5X100=100%
Completeness (EPA Method 6020A: Dissolved and Total Metals) =5/5X100=100%
Completeness (EPA Method SW9056A: Nitrate-N and Nitrite) =10/10X100=100%
Completeness (EPA Method 3520B/8270: 1,4-Dioxane) =1/1X100=100%

Completeness is affected by anything that reduces the number of samples analyzed (such as a sample loss during transport or extraction), as well as acceptance or non-acceptance of analytical results.

4.0 DATA VALIDATION

This data review covers thirteen water samples listed on page 10 including dilutions and reanalysis if applicable. The analyses were according to the following EPA Methods:

EPA Method **5030B/8260C** for VOCs by GC/MS
EPA Method **3520C/8270D** for SVOCs by GC/MS
EPA Method **8081B** for Organochlorine pesticides by GC/ECD
EPA Method **8082A** for Polychlorinated Biphenyls
EPA Method **8151B** for Chlorinated Herbicides
EPA Method **8015D (GROs),** Total Petroleum Hydrocarbons by GC/FID
EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID
EPA Method **8330B** for Nitrtoaromatics and Nitramine by HPLC/UV
EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV
EPA Method **6850** for Perchlorate by HPLC/MS
EPA Method **6020A** for Dissolved and total metals by ICP/MS
Method **7470A** for Dissolved and total Mercury by Cold Vapor
EPA **Method 9056A** for Nitrate and Nitrite by IC
EPA Method **3520C/8270 SIM** for 1,4-Dioxane by GC/MS

This review follows *Quality Assurance Project Plan, final Draft, USACE Fort Wingate Depot Activity*, McKinley County, New Mexico; Project # Eco-18-1237 April 2019, EM *200-1-10 Guidance for Evaluating Performance-based Chemical Data; US Army Corps of Engineers (USACE). June 2005,* and USEPA Analytical Operations/Data Quality Center (*AOC) National Functional Guidelines for Organic Data Review* (USEPA, January 2017); DoD QSM 5.3, 2019 and *National Functional Guidelines for Inorganic Data Review* (USEPA, September 2016). The Approved site-specific Quality Assurance Project Plan has the highest hierarchy.

The following subsections correlate to the above guidelines.

The followings are definitions of the data qualifiers:

U Indicates the analyses was analyzed for but not detected at or above Limit of Detection (LOD).

J	Indicates an estimated value with an unknown bias.
UJ	Indicates the analyte was analyzed for but not detected and reported less than LOD. However, the numerical value is approximate.
J^+	The result was estimated value and may be biased high.
J^-	The result was estimated value and may be biased low.
X	The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality criteria. The presence or absence of the analyte cannot be confirmed by the data provided. Acceptance or rejection of the data should be decided by the project team, but exclusion of the data is recommended

The following Reason codes were applied in the report:

M3	MS/MSD and/or LCS/LCSD percent recovery infraction with low bias
M4	MS/MSD or duplicate precision infraction
S1	Surrogate percent recovery infraction with high bias
S2	Surrogate percent recovery infraction with low bias
R4	Result exceeds calibration range
B6	Trip blank infraction (qualified detect)
B7	Field blank infraction (qualified detect)
B8	Equipment blank infraction (qualified detect)
D1	Field duplicate precision infraction

4.1. VOC (EPA Method 5030B/8260C)

4.1.1. Technical Holding Times

Holding time requirement was met for all samples. Five water samples were collected on 04-26-21. Samples were analyzed on 04-27-21 and 04-28-21 within holding time requirement. 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 04-15-21and at the beginning of each analysis shift on 04-27-21 and 04-28-21. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 04-15-21. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

Minimum average response factors for the system performance check compounds (SPCCs) were recognized according to the following table:

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 04-15-21
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

Average response factor curve fit was used mainly through the initial calibration. All target compounds met the maximum 15% RSD limit.

Least square linear regression was used for the following compound where %RSD exceeded the maximum 15 percent limit.

Target Analytes	Least Square Linear Regression (CCF) 04-15-21
2-Butanole	0.9995

Minimum average response factors for all target compounds were within method's recommended values, except for: Acetone (0.039) and 2-Butanone (0.069).

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 04-15-21
Vinyl chloride	-≤ 20%	√
1,1-Dichloroethene	-≤ 20%	√
Chloroform	-≤ 20%	√
1,2-Dichloropropane	-≤ 20%	√
Toluene	-≤ 20%	√
Ethyl benzene	-≤ 20%	√

√ denotes passing method acceptance limits

4.1.4. Calibration Verification and Continuing Calibration

The initial calibration was verified by a second source standard on 04-15-21. Percent difference (%D) and/or drift between initial calibration RRFs (average response factors) and the initial calibration verification response factors (RFs) were less than or equal to 20% for all target compounds. Continuing calibration check standard was analyzed at the beginning and end of analysis each analysis shift on 04-27-21 and 04-28-21. Prior to each continuing calibration, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

Minimum average response factors for the system performance check compounds (SPCCs) were all within the method limits according to the following table:

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 04-27-21 (I&II)	Continuing cal. Response factors 04-28-21 (I&II)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. The calculated % difference between RFs from continuing calibration and average response factors from initial calibration is summarized in Table 4.1.4.2 for continuing calibration reports presented with the data package.

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-27-21) I&II	%Deviation from Initial calibration (04-28-21) I&II
Vinyl chloride	≤ 20%	√	√
1,1-Dichloroethene	≤ 20%	√	√
Chloroform	≤ 20%	√	√
1,2-Dichloropropane	≤ 20%	√	√
Toluene	≤ 20%	√	√
Ethyl benzene	≤ 20%	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list, except for the compounds listed in the table below for each analysis shift. These minor differences should not affect data quality.

VOC Target Compounds	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (04-27-21)		%Deviation from Initial calibration (04-28-21)	
		I	II	I	II
2-Chloroethylvinyl ether	≤ 20%	32.0*	50.5*	47.6*	58.3*
Trichlorofluoromethane	≤ 20%	--	36.3*	--	23.8*
Methyl acetate	≤ 20%	--	24.8*	--	29.2*

*Outside acceptance limits

4.1.5. Quality Control: The QC 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 LCS/LCSD. Percent recoveries and percent RPDs for QC samples reported, were within the project acceptance limits for the entire compound list. The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blanks presented with the data package, analyzed with samples did not show presence of any target compounds.

Surrogate recoveries were all within the method's acceptable limits.

4.1.6. **Field duplicate sample** and its associated sample: No field duplicate sample was assigned to this method.

4.1.7. Raw data was submitted for all samples. Sample QC26042021EB5 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. Traces of Bromoform, Bromodichloromethane and Dibromochloromethane were detected in equipment blank. These compounds were not detected in any of the field samples. 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. Five water samples were collected on 04-26-21, extracted on 05-03-21 and were analyzed on 05-06-21 within holding time requirement.

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 03-18-20 and 03-19-20 and at the beginning of analysis shift on 05-06-21. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Due to long list of analytes used for this method, three separate lists of compounds were grouped together and initial calibration was generated separately for each group.

Initial calibration curve was generated on 03-18-20 and 03-19-20. 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 03-18-20
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

Average response factors for the rest of target compounds were within method's recommended values.

Calibration check compounds (CCCs) met the acceptance criteria for %RSD (less than 15%) among the response factors calculated for each level. Table 4.2.3.2 lists the CCCs with method requirement limits and calculated %RSD among response factors for initial calibration.

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Accepted Response Factors 03-18-20
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) 03-18-20
Benzoic acid	0.9985
2,4-Dinitrophenol	0.9990
4,6-Dinitro-2-Methylphenol	0.9982
Benzidine	0.9985
Dinoseb	0.9993
3,3-dimethylbenzidine	0.9987

All analytes met the acceptance criteria regarding minimum average response factor and maximum %RSD.

4.2.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 03-19-20. 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 05-06-21 and 05-07-21. Prior to continuing calibration, 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 (05-06-21) I	Continuing cal. Response factors (05-07-21) II
N-Nitroso-di-n-propylamine	≥ 0. 5	√	√
Hexachlorocyclopentadiene	≥ 0.05	√	√
2,4-Dinitrophenol	≥ 0.01	√	√
4-Nitrophenol	≥ 0.01	√	√

√ denotes passing method acceptance limits

Calculated percent differences (%D) between initial calibration RRFs (average response factors) and the continuing calibration response factors (RFs) were less than or equal to 20% for all the Calibration Check Compounds (CCCs) and less than or equal to 20% for nearly all other target analytes. Area counts for all internal standards were within ± 50 percent of the same level in the initial calibration. Percent difference between RFs from continuing calibration and average response factors from initial calibration passed the method's criteria as summarized in Table 4.2.4.2.

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (05-06-21) I	Accepted Deviation from Initial calibration (05-07-21) II
Phenol	≤ 20	√	√
1,4-Dichlorobenzene	≤ 20	√	√
2-Nitrophenol	≤ 20	√	√
2,4-Dichlorophenol	≤ 20	√	√
Hexachlorobutadiene	≤ 20	√	√
4-Chloro-3-methylphenol	≤ 20	√	√
2,4,6-Trichlorophenol	≤ 20	√	√
Acenaphthene	≤ 20	√	√
N-Nitrosodiphenylamine	≤ 20	√	√
Pentachlorophenol	≤ 20	√	√
Fluoranthene	≤ 20	√	√
Di-n-Octylphthalate	≤ 20	√	√
Benzo(a)pyrene	≤ 20	√	√

√ denotes passing method acceptance limits

Deviation from the initial calibration was less than 20 percent for the rest of target list except for the compounds listed in the table below:

Analytes	%Deviation from Initial calibration Method Criteria	%Deviation from Initial calibration (05-06-21) I	%Deviation from Initial calibration (05-07-21) II
Benzidine	≤ 20	24.1*	√
1,3,5-Trinitrobenzene	≤ 20	26.7*	√

*Exceeded 20% maximum allowed difference in the closing daily standard

This minor difference should not affect the quality of presented data.

4.2.5. Quality Control: The QC samples reported consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. The full list of target compounds was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits for the entire compound list in LCS/LCSD.

The results, 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 QC26042021EB5 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.

4.3. 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 (6) water samples were collected on 04-23-21 and 04-26-21, extracted on 04-29-21 and 05-03-21 and analyzed on 05-07-21 and 05-08-21, 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.

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 01-21-21 and before sample analysis 05-07-21 and 05-08-21. Breakdown of DDT to DDE and DDD and breakdown of Endrin-to-Endrin aldehyde and Endrin ketone were within the QC limits (less than 15 percent).

4.3.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each pesticide on 01-20-21 and 01-21-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was also generated for Toxaphene and Chlordane for both channels. %RSD among the calibration factors was less than 15 for Toxaphene and Chlordane.

Retention time window width were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.3.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene and Chlordane, on 01-20-21 and 01-21-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Performance of instrument was monitored by analysis of DDT and Endrin breakdown mixture. Before continuing (daily) calibration a mixture of DDT and Endrin was analyzed. Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of five continuing calibration standards were analyzed at 10-injections interval on 05-07-21 and 05-08-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds from both channels A and B, except for few compounds in channel B.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only. Therefore, this minor difference should not affect the quality of data.

In the five continuing calibration standards, one mid-point concentration of 20-40µg/L was injected.

4.3.4. Quality Control samples consisted of two method blanks and two sets of LCS/LCSD only. No sample was designated to be spiked for MS/MSD for this method. All pesticide target list was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Results for method blank were reviewed for each component and no organochlorine pesticide was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

4.3.5. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this method.

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4.3.6. Raw data was submitted for all samples. Sample QC26042021EB5 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.

4.4. 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 sample requested for this method. Three water samples were collected on 04-23-21 and 04-26-21, extracted on 04-29-21 and 05-03-21, and analyzed on 05-01-21 and 05-07-21 within required holding time.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.4.2. Initial Calibration

Two sets of initial calibration were used for reporting for this method. Initial calibrations were performed with seven concentration levels for Aroclor 1016 and Aroclor1260 on 03-10-21 and 05-06-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for each initial calibration.

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 03-10-21 and 05-06-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors were injected at single point for identification only.

A total of five continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-01-21 and 05-07-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for channels.

Results for surrogate recoveries and QC were all calculated from channel A. Channel B was used for confirmation only

4.4.4. Quality Control samples consisted of two method blanks and two sets of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. Percent recoveries (%R) for 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. Sample QC26042021EB5 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.

4.5. 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 sample requested for this method. Two water samples were collected on 04-26-21, extracted on 04-30-21 and analyzed on 05-26-21.

A dual column GC equipped with two Electron Capture Detectors (ECDs) was used for analysis. The results and raw data were submitted for both channel A and B. Channel A was used for data reporting.

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 05-19-21. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list.

Retention time windows were established for all target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.5.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 05-19-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-26-21, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all compounds in both channels.

4.5.4. 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. 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. Sample QC26042021EB5 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.

4.6. Nitroaromatics by HPLC/UV (EPA Method 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for four (4) water samples requested for this method. Water samples were collected on 04-26-21, extracted on 05-03-21 and analyzed on 05-19-21, within holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis. Positive results were confirmed with UHPLC equipped with different column (Kinetex- Biphenyl column).

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 04-29-21. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

Retention time windows were established for each target analyte at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 04-29-21. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of three continuing calibration standards were analyzed on 05-18-2 and 05-19-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

4.6.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No field sample was designated to be spiked as MS/MSD. All explosive target lists were spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.6.5 Field duplicate sample and its associated sample**:** No field duplicate sample was assigned to this method.

4.6.6. Raw data was submitted for all samples. Sample QC26042021EB5 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. All positive results were confirmed with confirmation column.

4.7. Nitroglycerine and PETN by UHPLC/UV (EPA Method 8332)

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for four (4) water samples requested for this method. Water samples were collected on

04-26-21, extracted on 05-03-21 and analyzed on 05-07-21 within required holding time.

A High-Performance LC (HPLC) coupled with Ultraviolet (UV) Detector was used for analysis.

4.7.2. Initial Calibration

Initial calibration was performed with five levels of concentration for each analyte on 10-27-20. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit
(Less than 15 %.)

Retention time windows were established for each target analytes at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.7.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each analyte on 10-27-20. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 05-07-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

4.7.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD only. No field sample was designated to be spiked as MS/MSD. Each target compound was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established acceptance QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. Surrogate recoveries were within the method QC acceptance limits.

4.7.5. Field duplicate sample and its associated sample**:** No field duplicate sample was assigned to this method.

4.7.6. Raw data was submitted for all samples. Sample QC26042021EB5 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.

4.8. Total Petroleum hydrocarbons GRO (EPA Method 8015G)

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Three (3) water samples were collected on 04-26-21. Samples were extracted and analyzed on 04-29-21 within holding time requirement.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Sample was carried through the system by purge and trap.

4.8.2. Initial Calibration

Initial calibration was performed with six levels of concentration on 03-16-21. Calibration factor (area for each compound/concentration) was used to quantify gasoline range hydrocarbons (TPH as GRO). Average response factor was used to show linearity. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15%.)

Retention time window width was established by analysis of window defining hydrocarbon standard (C6-C10). Retention times for further sample analyses was used for peak identification and integration range.

4.8.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 03-16-21. 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. A total of three daily calibrations were carried out on 04-29-21 bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each group of GRO Hydrocarbons from continuing calibrations were less than 20%.

4.8.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. 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.

4.8.5. 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 QC26042021EB5 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.

4.9. Total Petroleum hydrocarbons DRO (EPA Method 8015D)

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water sample requested for this method. Three (3) water samples were collected on 04-26-21, extracted on 05-03-21 and analyzed on 05-04-21.

A GC coupled with Flame Ionization Detector (FID) was used for analysis. Heavier range of total petroleum hydrocarbons were extracted and introduced into system by direct injection.

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 07-01-20. 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 for surrogates only. Retention times for further sample analyses was used for peak identification and integration range.

4.9.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 07-01-20. 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. A total of three daily calibration standards were carried out on 05-04-21, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each DRO group from continuing calibrations were less than 20%.

4.9.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked for MS/MSD. Percent recoveries (%R) of LCS/LCSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no contamination was found in the method blank. Surrogate recoveries were all within the method QC acceptance limits.

4.9.5. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this method.

4.9.6. Raw data was submitted for all samples. Sample QC26042021EB5 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.

4.10. Perchlorate by HPLC/MS (EPA Method 6850)

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for all sample requested for this method. Five (5) water samples were collected on 04-26-21. Samples were analyzed on 05-04-21 within holding time.

A High-Performance LC coupled with Mass Detector (HPLC/MS) was used for analysis.

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 05-03-21. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($Cl^{18}O4^{-}$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9998 (Perchlorate ion 83) and 0.9997 (perchlorate ion 85) was calculated to show the linearity of each curve. The concentrations used for calibration ranged from 0.1 – 7.5 µg/L.

Retention time for each isotope (ion 83 and 85) at each calibration level was within 0.2 minutes required by the method.

4.10.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 05-03-21. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of four daily standards were carried out on 05-04-21, bracketing the analyses of samples and all the QC samples. Recoveries of continuing calibration standards were within 85-115% limit.

4.10.4. Quality Control samples consisted of one method blank and one set of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Each target was spiked and reported for LCS/LCSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed for each compound and no target was found in the method blank. No surrogate is used in this method.

4.10.5. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this method.

4.10.6. Raw data was submitted for all samples. Sample QC26042021EB5 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.

4.11. TOTAL and DISSOLVED METALS BY ICP (EPA Method 6020A)

4.11.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for five water samples requested for this method. Water samples were collected on 04-26-21. Samples were prepared (digested) on 04-30-21 for both total and dissolved metals. Samples and QC samples were analyzed on 05-19-21 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of analysis day on 05-19-21. Tune performance report was generated at the start of analysis day, before initial calibration. It was within method’s acceptance criteria. The initial instrument (ICP-MS) calibration for this method was acceptable. One method blank and one calibration standard were used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

Metals	**Concentration µg/L**
Se, Pb, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Mn, Ni, Ag, Tl, V, and Zn	50,100,500,1000
Al, Fe, Ca, Mg, Na, and K	50,000

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of ±20% of the true value for the analytes incorporated in each solution.

4.11.3. Quality Control samples consisted of one method blank and one set of LCS/LCSD for total and dissolved metals. No sample was designated to be spiked as MS/MSD for total and/or dissolved metals. 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 each method blank. Calibration blanks were analyzed after each continuing calibration standard.

4.11.4. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this method.

4.11.5. Raw data was submitted for all samples. Sample QC26042021EB5 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.

4.12. MERCURY by COLD VAPOR (Dissolved and total): EPA Method 7470A

4.12.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for five (5) water samples requested for this method. Water samples were collected on

04-26-21. Samples were prepared (digested) and analyzed on 05-13-21 for Mercury. Samples were prepared on 05-05-21 and analyzed on 05-06-21 for dissolved Mercury. The samples were preserved and filtered in the lab for dissolved Mercury analysis.

4.12.2. Initial and continuing calibrations: The instrument calibration for this method was acceptable. One blank and five standard levels were used for calibration curve at the beginning of each analysis day on 05-06-21 and 05-13-21. The correlation coefficient of at least 0.999 was calculated to show the linearity of each initial calibration curve. The concentrations used for calibration ranged from 0.2 – 5.0 µg/L.

Initial calibration verification and Continuing calibration verification standards was within the acceptable range (90-110% of the spiked value) for each calibration curve.

4.12.3. Quality Control: The data for all the QC samples were within acceptable control limits. The QC samples consisted of one method blank and LCS/LCSD for total and dissolved Mercury. No sample was designated to be spiked as MS/MSD. Percent recoveries and % differences were within the control limits for LCS/LCSD for total and dissolved Mercury. Recoveries of LCS/LCSD were within acceptable range of 82-119%. The results were all within QC acceptable limits. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: No field duplicate sample was assigned to this method.

4.12.5. Raw data was submitted for all samples. Sample QC26042021EB5 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.

4.13. Method SW9056A: Nitrate-N and Nitrite

4.13.1. Technical Holding Times: Holding time from sample collection to analysis was met for all water samples requested for this method. A total of ten (10) water samples were collected on

04-26-21, and were analyzed on 04-27-21 and 04-28-21 within the required 48-hour holding time.

4.13.2. Initial and continuing calibrations: Anions such as Chloride, Fluoride, Nitrite, Nitrate and Sulfate were separated from water samples by Ion chromatography. The separated anions in their acid form (very conductive) were measured by conductivity. They were identified on the basis of retention time as compared to reference standards.

The instrument was initially calibrated with nine calibration levels (0.05-20mg/L) on 03-29-21. A second instrument was also used for sample analysis (calibrated on 04-23-2). Linear curve type with correlation coefficient of at least 0.999 was used throughout analysis. Percent RSD among calibration factors was less than 15%. Calibration curve (concentration versus area count of each anion) was presented for each component. Area for each level was randomly checked with the values used in each calibration curve. All agreed with the raw data. A second source standard mixture (ICV) was used to verify the linearity of each initial calibration on 03-30-21 and 04-23-21. Recoveries were all within 90-110% of initial value. Continuing Calibration standards at 10-injections interval were analyzed on 04-27-21 and 04-28-21. A total of nine continuing calibration standards were analyzed with re-analysis of some anions. In all continuing calibration standards submitted, the recoveries of target anions were within 90-110% of the expected values. After each continuing calibration standard, one calibration blank was injected. All the blanks were reported as non-detected regarding all the target anions. Retention time window width was established and confirmed in each 24-hour. It was within the assigned QC limit for each anion.

4.13.3. Quality Control Samples consisted of two method blanks and two sets of LCS/LCSD. No sample was designated to be spiked as MS/MSD. Recoveries of LCS/LCSD were within 90-110 % of spiked values for Nitrate and Nitrite. Percent RPDs were less than 20% for LCS/LCSD.

4.13.4. Field duplicate sample and its associated sample: Sample TMW25042021 was identified as field duplicate of sample TMW25042021D.

Results for sample and corresponding field duplicate sample are shown in the table below for target anions:

	TMW25042021 mg/L	TMW25042021 mg/L	%RPD
Nitrate-N	0.31	0.31	≤1
Nitrite-N	U	U	--

4.13.5. Raw data was submitted for all samples. Sample QC26042021EB5 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.

4.14. 1,4-Dioxane (EPA Method 3520C/8270SIM)

4.14.1. Technical Holding Times

Holding time requirement was met for this sample. One water sample was collected on 04-26-21, extracted on 04-28-21 and were analyzed on 04-29-21; within holding time.

Selective Ion Monitoring (SIM) method with GC/MS was used for analysis.

Isotopically-labeled 1,4-Dioxane-d8 was added to serve as internal standard.

The chain-of-custody was reviewed for documentation of sample information and method of analysis.

Qualification notations, if any, will be summarized in result section; section 4.14.7.

4.14.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 04-29-21 and at the beginning of analysis batch on 04-29-21. It passed all the method assigned criteria.

4.14.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD.

Initial calibration curve was generated on 04-29-21. A multi-level calibration standard ranging from 0.05mg/L to 50mg/L was used for this purpose. Internal standard curve type in

Selective Ion Monitoring (SIM) mode was used for initial calibration and all following analysis. 1,4-Dioxane-d8 was used as internal standard. Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table. Average response curve type was used for 1,4-Dioxane and Bromobenzene as surrogate. Target compounds met the maximum 15% RSD limit (all less than 15%).

4.14.4. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 04-29-21. Percent difference (%D) were within ±20% of the expected value.

Continuing calibration check standard was analyzed at the beginning and at the end of analysis shift on 04-29-21. Prior to opening continuing calibration standard, instrument performance tune check standard (BFB) was carried out. It passed all the method tuning criteria. Percent difference (%D) and/or drift for both opening and closing continuing calibration standards were within ±20% of the expected value for each daily standard.

4.14.5. Quality Control: The QC samples reported consisted of one method blank and one set of LCS/LCSD only. No sample was designated to be spiked as MS/MSD. 1,4-Dioxane as the only target with Bromobenzene as surrogate was spiked and reported for LCS/LCSD. Percent recoveries and percent RPDs for all the QC samples reported were within the project acceptance limits.

The results, percent recoveries and RPDs were recalculated randomly and all agreed with the reported QC summary table. Method blank presented with the data package, analyzed with samples did not show presence of any target compound.

Surrogate recoveries were all within method’s acceptance limit.

4.14.6. **Field duplicate sample** and its associated sample: No field duplicate sample was assigned to this method.

4.14.7. Sample QC26042021EB5 was selected to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #21D296 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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