

Final Data Validation Report

USACE Fort Wingate Depot Activity New Mexico

Project No: Eco-18-1237

SDG #23J027 Analytical Data Package

Publication Date: 01-10-2024

By
ZIBA HOSSEINI
14343 Peach Hill Rd.
Moorpark, CA, 93021

TABLE OF CONTENTS

EXECUTIVE SUMMARY	4
1.0 INTRODUCTION	
1.1 Objectives and Scope of the Data Validation	6
1.2 Organization of the Report	6
2.0 DATA REVIEW AND VALIDATION	
2.1 Data Reporting	7
2.2 Data Evaluation	8
2.2.1 Holding Times	15
2.2.2 Laboratory and Field Blanks	16
3.0 QUALITY ASSURANCE OBJECTIVES	
3.1 Qualitative QA Objectives	17
3.1.1 Comparability	17
3.1.2 Representativeness	17
3.2 Quantitative QA Objectives	17
3.2.1 Precision	18
3.2.2 Accuracy	18
3.2.3 Completeness	19
4.0 DATA VALIDATION	
4.0 Results of Data Validation	20
4.1. VOCs by EPA Method 8260C	21
4.2. Semivolatiles + Extra analytes by EPA Method 8270D	25
4.3. Organochlorine Pesticides by EPA Method 8081B	30
4.4. Polychlorinated Biphenyls by EPA Method 8082A	32
4.5. Chlorinated Herbicides EPA Method 8151A	34
4.6. Nitroaromatics & Nitramines by EPA Method SW846 3535A/8330B	36
4.7. Nitroglycerine and PETN by EPA Method 8332	37
4.8. Total Petroleum Hydrocarbons (GROs) By EPA Method 8015D	38
4.9. Total Petroleum Hydrocarbon (DROs) by EPA Method 8015D	40

4.10.	Perchlorate by EPA Method 6850	41
4.11.	Total and Dissolved Metals by ICP-MS (EPA Method 6020A)	43
4.12.	Mercury & Dissolved Mercury by EPA Method 7470A	45
5.0	CONCLUSION	46
6.0	REFERENCES	47

EXECUTIVE SUMMARY

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

A total of thirteen (13) water samples were collected on 10-02-23. EMAX Laboratories received the samples on 10-04-23. Data was delivered in one package as stage 2b and stage 3 deliverable. Ten percent of the data was subjected to validation equivalent to stage 3 deliverable. Raw data for all samples were submitted for the requested analytical methods. One sample from this sample delivery group, BGMW12102023 (Lab ID#J027-05) was reviewed as stage 3 data deliverable. Raw data for this sample was compared to the reported summary tables for each method and went through comprehensive data validation review. No sample was designated to be spiked as MS/MSD on the chain of custody. Therefore, recoveries and results of LCS/LCSD was used both for accuracy and precision. Raw data for method blank and LCS/LCSD for each method were cross checked with the corresponding summary table results.

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

EPA Method 3050B/8260C: Volatile Organics by GC-MS (13 samples)

EPA Method 3520C/8270D: Semi-Volatile Organics by GC/MS (12 samples)

EPA Method SW846 3535A/8330B: Nitroaromatics and Nitramines (12 samples)

EPA Method SW8332: Nitroglycerine and PETN (12 samples)

EPA Method 8081B: Organochlorine Pesticides (8 samples)

EPA Method 8082A: Polychlorinated Biphenyls; PCBs (3 sample)

EPA Method 8151A: Chlorinated herbicides (3 sample)

EPA Method 8015D: Total Petroleum Hydrocarbons (GROs) (4 samples)

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

EPA Method 6850: Perchlorate (12 samples)

EPA Method SW6020A: Dissolved and total Metals by ICP-MS (12 samples)

EPA Method 7470A: Mercury & Dissolved Mercury (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. However, two samples (BGMW12102023 and MW32102023) were extracted four days past holding time for Herbicides. The deviations, if any, are discussed in Section 4.0 for each method.

The SDG # 23J027 analytical data evaluated in this data validation report has met the data quality and usability requirement as defined in the data quality objectives. While very few analytical QC exceedances were observed, it was not significant for any data qualifiers. Overall data is of acceptable quality and considered usable for its intended purpose.

1.0 INTRODUCTION

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

1.1 Objectives and Scope of Data

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

1.2 Organization of the Report

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

2.0 DATA REVIEW AND VALIDATION

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

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

A total of thirteen (13) water samples were collected on 10-02-23. EMAX Laboratories received the samples on 10-04-23.

2.1 Data Reporting

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

EMAX Laboratories provided the following information in one data package:

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

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

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

Stage 3 review compared the reported analytical results with those obtained from the raw data. Raw data for each analytical method requested on the chain of custody were submitted for all samples. One field sample BGMW12102023 (EMAX ID #J027-05) from this sample delivery group was reviewed as stage 3 data review. Raw data for this sample was evaluated comprehensively. No sample was designated to be spiked as MS/MSD on the chain of custody. Raw data for method blank and LCS/LCSD was reviewed for each method in detail. Calculations and corresponding equations, as well as analyte identification were randomly checked and verified.

2.2 Data Evaluation

The following parameters were evaluated in the preliminary data review:

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

- Technical holding times were confirmed for all samples with regard to the requested method of analysis (collection to extraction and extraction to analysis);
- Limit of quantitation (LOQ) for each analyte reported were compared with the project measurement objectives;
- Initial calibration and initial calibration verification standards were evaluated;
- Continuing calibration standards were evaluated
- Trip blank results (Method 8260C and TPH by purge & trap only) were evaluated;
- MS/MSD results and recoveries were evaluated; only for total metals
- LCS/LCSD results and recoveries were evaluated; and
- Method blank results as well as surrogate recoveries, internal standards, 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#23J027			Matrix: Water	
Field/Client ID	Lab ID	Date collected	Validation stage	Requested Methods of Analysis
MW23102023	23J027-01	10-02-23	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
TMW44102023	23J027-02	10-02-23	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
TMW16102023	23J027-03	10-02-23	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
FDUP01-102023	23J027-04	10-02-23	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850

Site Name: Fort Wingate, New Mexico				
SDG#23J027			Matrix: Water	
Field/Client ID	Lab ID	Date collected	Validation	Requested Methods of Analysis
BGMW12102023	23J027-05	10-02-23	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 TPH Gasoline; TPH as DRO
TMW46102023	23J027-06	10-02-23	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
TMW19102023	23J027-07	10-02-23	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
MW32102023	23J027-08	10-02-23	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Dissolved Mercury Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850 TPH Gasoline; TPH as DRO

Site Name: Fort Wingate, New Mexico				
SDG#23J027			Matrix: Water	
Field/Client ID	Lab ID	Date collected	Validation stage	Requested Methods of Analysis
BGMW03102023	23J027-09	10-02-23	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
TMW18102023	23J027-10	10-02-23	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
BGMW08102023	23J027-11	10-02-23	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 Chlorinated Herbicides Polychlorinated Biphenyls Perchlorate by 6850

Site Name: Fort Wingate, New Mexico SDG#23J027				
				Matrix: Water
Field/Client ID	Lab ID	Date collected	Validation stage	Requested Methods of Analysis
TMW36102023	23J027-12	10-02-23	S3VM	VOCs by SW5030B/8260C, Semivolatiles + APP9 Organochlorine Pesticides Nitroaromatics and Nitramines Nitroglycerine & PETN Mercury Dissolved Mercury Dissolved Metals by ICP-MS Total Metals by ICP-MS Perchlorate by 6850
QC02102023TB	23J027-13	10-02-23	S3VM	VOCs by SW5030B/8260C, TPH Gasoline;

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

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

MATRIX	CONSTITUENT	EPA METHOD	LOQ
Water	Volatile Organic Compounds list	SW5030B/8260C	1,2 & 20 µg/L
	Semi Volatile Organic Compound List	SW3520C /8270D	10&20µg/L, (Benzidine=40µg/L)
	Nitroaromatics & Nitramines	SW3535A/8330B	0.4µg/L
	Nitroglycerine & PETN	SW3535A/8332B	0.4µg/L
	Chlorinated Herbicides	SW8151A	1µg/L, (MCPA=40µg/L)
	Organochlorine Pesticides	SW8081B	0.1µg/L Methoxychlor =1.0µg/L Toxaphene =2.0µg/L
	Polychlorinated Biphenyls (PCBs)	SW8082A	1µg/L
	Total Petroleum Hydrocarbons (GROs)	SW8015D Purge & Trap	100µg/L
	Total Petroleum Hydrocarbons (DROs)	SW8015D Extractable	0.5mg/L
	Dissolved & Total Metals By ICP-MS	SW6020A	0.5µg/L, 1µg/L, 20µg/L, 100µg/L, 200µg/L
	Dissolved Mercury/Mercury	SW7470A	0.5µg/L
	Anions by IC	SW9056A	0.1mg/L; 0.2mg/L; 0.5mg/L
	Perchlorate	SW6850	0.5µg/L

2.2.1 Sample Receipt

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

2.2.2 Holding Times

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

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

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

*Two samples (BGMW12102023 and MW32102023) were extracted four 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 thirteen ice preserved coolers and were stored in a refrigerator upon arrival to the laboratory. The cooler's temperature was reported as low as 1.2°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

R₁ = Result of the first duplicate or measured sample concentration

R₂ = Result of the second duplicate or known sample or duplicate concentration

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

Precision results are discussed in Section 4.0 of this report.

3.2.2 Accuracy

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

3.2.3 Completeness

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

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

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

Completeness (EPA Method 5030B/8260C: VOCs) = 13/13X100=100%

Completeness (EPA Method 3520B/8270D: SVOCs) = 12/12X100=100%

Completeness (EPA Method 3535A/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) = 3/3X100=100%

Completeness (EPA Method 8151B: Chlorinated Herbicides) = 3/3X100=100%

Completeness (EPA Method 8015G: Petroleum Hydrocarbons; GRO) = 4/4X100=100%

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

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

Completeness (EPA Method 7470A: Mercury & Dissolved Mercury) = 12/12X100=100%

Completeness (EPA Method 6020A: Dissolved and Total Metals) = 12/12X100=100%

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

4.0 DATA VALIDATION

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

EPA Method **5030B/8260C** for VOCs by GC/MS

EPA Method **3520C/8270D** for SVOCs by GC/MS

EPA Method **8081B** for Organochlorine pesticides by GC/ECD

EPA Method **8082A** for Polychlorinated Biphenyls

EPA Method **8151B** for Chlorinated Herbicides

EPA Method **8015D (GROs)**, Total Petroleum Hydrocarbons by GC/FID

EPA Method **8015D (DROs)**, Total Petroleum Hydrocarbons by GC/FID

EPA Method **3535A/8330B** for Nitroaromatics and Nitramine by HPLC/UV

EPA Method **8332** for Nitroglycerine and PETN by UHPLC/UV

EPA Method **6850** for Perchlorate by HPLC/MS

EPA Method **6020A** for Dissolved and total metals by ICP/MS

Method **7470A** for Mercury & Dissolved Mercury by Cold Vapor

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 thirteen samples. Water samples were collected on 10-02-23. Samples were analyzed on 10-12-23 and 10-13-23 within method's requirement for holding time. (Water samples were preserved with hydrochloric acid).

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

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

4.1.2. Tuning criteria

Performance of the instrument was checked by injection of a single component tune check standard (BFB: Bromofluorobenzene) prior to initial calibration on 10-05-23 and at the beginning of analysis shift on 10-12-23. It passed all the method assigned criteria.

4.1.3. Initial Calibration

Samples were analyzed with reference to one set of initial calibration using GC/MSD. Initial calibration curve was generated on 10-05-23. A multi-level calibration standard ranging from 0.3µg/L to 100µg/L was used for this purpose. Internal standard curve type was used for initial calibration and all following analysis. Minimum response factor for system performance check compounds (SPCCs) were within the method acceptable limits (Table 4.1.3.1). Response factors at each level were randomly recalculated and all agreed with the response factors submitted in the initial calibration summary table.

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

Table 4.1.3.1: System Performance Check Compounds (Initial calibration)

System Performance check compounds (SPCCs)	Minimum average response factor (requirement)	Average Response factor 10-05-23
Chloromethane	≥ 0.10	√
1,1-Dichloroethane	≥ 0.20	√
Bromoform	≥ 0.10	√
Chlorobenzene	≥ 0.50	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√

√ denotes passing method acceptance limits

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

Minimum average response factors for all target compounds were within method's recommended values, except for: 2-Butanone (0.048). However, recoveries were within the requirement of 70-130%. Calibration check compounds (CCCs) met the acceptance criteria for

%RSD among the response factors calculated for each level. Table 4.1.3.2 lists the CCCs with method requirement limits for %RSD among response factors for initial calibration.

Table 4.1.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	Response Factors %RSD 10-05-23
Vinyl chloride	≤ 20%	√
1,1-Dichloroethene	≤ 20%	√
Chloroform	≤ 20%	√
1,2-Dichloropropane	≤ 20%	√
Toluene	≤ 20%	√
Ethyl benzene	≤ 20%	√

√ denotes passing method acceptance limits

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

Target Analytes	Least Square Linear Regression (CCF) 10-05-23
Chloromethane	0.999
Bromomethane	0.9938
Methylene Chloride	0.9989
Chloroform	0.9993
Bromodichloromethane	0.9980

4.1.4. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard on 10-05-23. 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 standard was analyzed at the beginning and end of analysis shift on 10-12-23 and 10-13-23. Prior to continuing calibration standard, **instrument performance check standard** (BFB tune check) was carried out. It passed all the method tuning criteria.

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

Table 4.1.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors 10-12-23 (opening)	Continuing cal. Response factors 10-13-23 (closing)
Chloromethane	≥ 0.10	√	√
1,1-Dichloroethane	≥ 0.20	√	√
Chlorobenzene	≥ 0.50	√	√
Bromoform	≥ 0.10	√	√
1,1,2,2-Tetrachloroethane	≥ 0.30	√	√

√ denotes passing method acceptance limits

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

Table 4.1.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	%Deviation From Initial calibration (Acceptance Limit)	%Deviation from Initial calibration (10-12-23) opening	%Deviation from Initial calibration (10-13-23) closing
Vinyl chloride	$\leq 20\%$	√	√
1,1-Dichloroethene	$\leq 20\%$	√	√
Chloroform	$\leq 20\%$	√	√
1,2-Dichloropropane	$\leq 20\%$	√	√
Toluene	$\leq 20\%$	√	√
Ethyl benzene	$\leq 20\%$	√	√

√ denotes passing method acceptance limits

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

Target Compounds	% Deviation From Initial calibration (Acceptance Limit)	Deviation from Initial calibration (10-12-23) opening	Deviation from Initial calibration (10-13-23) closing
Naphthalene	≤ 20	26.3*	√
Bromomethane	≤ 20	√	27.5*
Carbontetrachloride	≤ 20	√	25.9*

*Outside control limit

However, recoveries were all within 50% of the expected value in the closing daily standard.

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

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

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

4.1.6. Field duplicate sample and its associated sample: Field sample FDUP01-102023 was identified as field duplicate of sample TMW16102023. No VOCs was detected in any of field sample or associated field duplicate sample.

4.1.7. Raw data was submitted for all samples. Sample BGMW12102023 (Lab ID# 23J027-05) was reviewed as stage 3 data deliverable. Raw data for this sample with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data agreed with all the results reported in data summary reports.

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

4.2.1. Technical Holding Times

Holding time requirement was met for all samples. Twelve water samples were collected on 10-02-23, extracted on 10-09-23 and were analyzed on 10-11-23 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 each set of initial calibration on 07-10-23 and at the beginning of analysis shift on 10-11-23. It passed all the method assigned criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

4.2.3. Initial Calibration

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

Initial calibration curves were generated on 07-10-23. 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 07-10-23
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

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

Table 4.2.3.2 Calibration Check Compounds (CCCs) Initial Calibration

Calibration Check Compounds (CCCs)	Response Factors %RSD (Method limit)	%RSD Response Factors 07-10-23
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) 07-10-23
Benzoic acid	0.9994
4,6-Dinitro-2-Methylphenol	0.9968
Butyl benzyl phthalate	0.9997
Di-n-octylphthalate	0.9997

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

4.2.4. Initial Calibration Verification and Continuing Calibration

Each set of initial calibration was verified by a second source standard on 07-10-23. 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 Benzoic acid (%D=25.1) and 4,6-Dinitro-2-Methylphenol (%D=27.3).

Continuing calibration standards were analyzed at the beginning and at the end of analysis shift on 10-11-23. Prior to continuing calibration injection, instrument performance tune check standard (DFTPP) was carried out. It passed all the method tuning criteria. Tailing of Pentachlorophenol and Benzidine was within acceptance limit (less than 2%) and breakdown of DDT was also within methods acceptance limit of less than 20%.

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

Table 4.2.4.1: System Performance Check Compounds (Daily calibration)

System Performance Check compounds (SPCCs)	Minimum response factor (Method limits)	Continuing cal. Response factors (10-11-23) I&II
N-Nitroso-di-n-propylamine	≥ 0.5	√
Hexachlorocyclopentadiene	≥ 0.05	√
2,4-Dinitrophenol	≥ 0.01	√
4-Nitrophenol	≥ 0.01	√

√ denotes passing method acceptance limits

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

Table 4.2.4.2 Calibration Check Compounds (CCCs): Continuing Calibration

Calibration Check Compounds (CCCs)	% Deviation From Initial calibration (Acceptance Limit)	Accepted Deviation from Initial calibration (10-11-23) I& II
Phenol	≤ 20	√
1,4-Dichlorobenzene	≤ 20	√
2-Nitrophenol	≤ 20	√
2,4-Dichlorophenol	≤ 20	√
Hexachlorobutadiene	≤ 20	√
4-Chloro-3-methylphenol	≤ 20	√
2,4,6-Trichlorophenol	≤ 20	√
Acenaphthene	≤ 20	√
N-Nitrosodiphenylamine	≤ 20	√
Pentachlorophenol	≤ 20	√
Fluoranthene	≤ 20	√
Di-n-Octylphthalate	≤ 20	√
Benzo(a)pyrene	≤ 20	√

√ denotes passing method acceptance limits

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

Target Compounds	% Deviation From Initial calibration (Acceptance Limit)	Deviation from Initial calibration (10-11-23) opening	Deviation from Initial calibration (10-11-23) closing
Butyl Benzylphthalate	≤ 20	21.0*	23.5*
Di-n-octylphthalate	≤ 20	21.9*	23.2*
4,6-Dinitro-2-methyl phenol	≤ 20	√	34.1*

*Outside control limit

However, recoveries were all within 50% of the expected value in the closing daily standard.

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

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

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 two surrogates in samples shown in the table below:

Surrogate	MW23102023 (J027-01)	FDUP01-102023 (J027-04)	TMW46102023 (J027-06)	BGMW08102023 (J027-11)	QC Limit%
2-Fluorophenol	√	15%*	√	15%*	19-119
Phenol-D5	39%*	28%*	38%*	33%*	40-130

*Outside QC limit

This was attributed to matrix interference in the case narrative.

4.2.6. Field duplicate sample and its associated sample: Field sample FDUP01-102023 was identified as field duplicate of sample TMW16102023. No SVOCs was detected in any of field sample or associated field duplicate sample.

4.2.7. Raw data was submitted for all samples. Sample BGMW12102023 (Lab ID# 23J027-05) was reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.3.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for eight water samples requested for this method. Water samples were collected on 10-02-23, extracted on 10-06-23 and analyzed on 10-13-23, and 10-24-23 within required holding time.

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

DDT- Endrin breakdown mix was analyzed prior to initial calibration on 10-12-23

and before sample analysis on 10-13-23 and 10-24-23. 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 10-12-23. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 15%). Pesticide target list was calibrated with two separate groups of compounds for each column. Due to interference with other pesticide target compounds, a separate curve was generated for Toxaphene on 10-11-23 for both channels. %RSD among the calibration factors was less than 15 for Toxaphene.

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

4.3.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all pesticide target list including Toxaphene, on 10-11-23 and 10-12-23. 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 running each continuing (daily) calibration standard, a mixture of DDT and Endrin was analyzed. Breakdown of DDT to DDE and DDD and breakdown of Endrin to Endrin-aldehyde and Endrin-ketone were all less than 15%.

A total of four continuing calibration standards were analyzed at 10-injections interval on 10-11-23 and 10-24-23, 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 for both channels A and B. In all continuing calibration standards, one mid-point concentration of 20-40µg/L was injected. Results for surrogate recoveries and QC were all calculated from channel B. Channel A was used for confirmation only.

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

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

Surrogate recoveries were all within the method QC acceptance limits.

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

4.3.6. Raw data was submitted for all samples. Sample BGMW12102023 (Lab ID# 23J027-05) was reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.4.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Three water samples were collected on 10-02-23, extracted on 10-06-23 and analyzed on 10-12-23.

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 Aroclor 1260 on 05-10-23. 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 was established at each calibration level. Retention times for further sample analyses were within the assigned retention time windows set by initial calibration.

4.4.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for Aroclor 1016 and 1260, on 05-10-23. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels. After establishing linearity of the instrument through initial calibration, the rest of Aroclors, if required, were injected at single point for identification only.

Two continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-12-23, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for each channel.

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

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

Results for method blank was reviewed and no PCBs 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 analysis was assigned to this method.

4.4.6. Raw data was submitted for all samples. Sample BGMW12102023(Lab ID#23J027-05) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.5. Chlorinated Herbicides (EPA 8151A)

4.5.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for water samples requested for this method. Three water samples were collected on 10-02-23, extracted on 10-05-23 and 10-11-23 within holding time. Due to low surrogate recoveries, two samples were extracted four days past holding time. Samples were analyzed on 10-11-23 and 10-17-23.

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

4.5.2. Initial Calibration

Initial calibration was performed with eight levels of concentration for each herbicide on 09-18-23. Both channels A and B were calibrated. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) for both channels A and B were within method specification (less than 20%) for all target list. Linear regression curve type with correlation coefficient of 0.99901 was used for MCP in column B.

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

4.5.3. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for all target herbicides on 09-19-23. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20% for both channels.

five continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-11-23, 10-12-23 and 10-17-23, bracketing the analyses of sample and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations, were less than 20% for all target compounds in both channels.

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

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

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

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

4.5.6. Raw data was submitted for all samples. Sample BGMW12102023 (Lab ID#23J027-05) with all related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports. As mentioned before, two samples (BGMW12102023 and MW32102023) were extracted four days past holding time. Initially, all samples were extracted and analyzed within holding time, but due to low surrogate recovery, these samples were re-extracted on 10-13-23, four days past holding time. Therefore, results reported for these samples will be qualified as estimated value, "UJ".

4.6. Nitroaromatics by LC/MS/MS (EPA Method 3535A/ 8330B)

4.6.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 10-02-23, extracted on 10-05-23 and analyzed on 10-05-23 and 10-06-23, within holding time.

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

4.6.2. Initial Calibration

Initial calibration was performed with seven concentration levels for each analyte on 08-29-23 and 08-30-23. Calibration factor (area for each compound/concentration) was used to quantify analytes. Average response factor was used to show linearity for both channels. Percent relative standard deviation (% RSDs) among calibration factors (CFs) was within acceptable limit (less than 15 percent.)

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

4.6.3. Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) for each target analyte on 08-30-23. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15% in both columns.

Continuing calibration standards were analyzed at 10-injections interval. A total of three continuing calibration standards were analyzed on 10-05-23 and 10-06-23, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for all analytes for both columns.

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

reported for LCS/LCSD. Percent recoveries (%R) were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

Result for method blank was reviewed and no target was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits. 3,4-Dinitrotoluene was used as surrogate.

4.6.5 Field duplicate sample and its associated sample: Field sample FDUP01-102023 was identified as field duplicate of sample TMW16102023. No explosives were detected in the field sample or associated field duplicate sample.

4.6.6. Raw data was submitted for all samples. Sample BGMW12102023(Lab ID#23J027-05) was reviewed as stage 3 data deliverable. Raw data for this sample together with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.7.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 10-02-23, extracted on 10-05-23 and analyzed on 10-06-23 within 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 09-01-23. 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 09-01-23. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 15%.

A total of three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-06-23, bracketing the analyses of samples and all the QC samples. Percent difference between initial calibration average response factors and the response factors calculated for each analyte from continuing calibrations were less than 15% for each analyte.

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

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

4.7.5. Field duplicate sample and its associated sample: Sample FDUP01-102023 was identified as field duplicate of sample TMW16102023. No explosive target compound was detected in sample or associated field duplicate sample.

4.7.6. Raw data was submitted for all samples. Sample BGMW12102023 (Lab ID#23J027-05) was reviewed as stage 3 data deliverable. Raw data of this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.8.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for four water samples requested for this method. Water samples were collected on 10-02-23. Samples were analyzed on 10-06-23 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 05-01-23. 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. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 05-01-23. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-06-23, 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 assigned to be spiked as MS/MSD. Percent recoveries (%R) were within the established QC limits for LCS/LCSD. Raw data for both un-spiked sample and spiked QC samples were matching the reported values. Result for method blank was reviewed and no contamination was found in the method blank.

Surrogate recoveries were all within the method QC acceptance limits.

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

4.8.6. Raw data was submitted for all samples. Sample BGMW12102023(Lab ID#23J027-05) was reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.9.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for three water samples requested for this method. Water samples were collected on 10-02-23, extracted on 10-05-23 and analyzed on 10-06-23 and 10-07-23 within holding time.

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

4.9.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 02-17-23. 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. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 02-17-23. Percent difference between initial calibration response factors (Average response factors) and the response factors calculated for each analyte were less than 20%

Three continuing calibration standards were analyzed at 10-injections interval. It was carried out on 10-06-23 and 10-07-23 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 two set of LCS/LCSD. No sample was designated for MS/MSD. Percent recoveries (%R) of LCS/LCSD were within the QAPP established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

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

4.9.6. Raw data was submitted for all samples. Sample BGMW12102023(Lab ID#23J027-05) was designated to be reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.10.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve samples requested for this method. Water samples were collected on 10-02-23. Samples were analyzed on 10-09-23 and 10-10-23, within holding time.

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

4.10.2. Initial Calibration

Initial calibration was performed with seven levels of concentration on 09-26-23. Internal standard curve type was used for quantifying Perchlorate. Isotopically-labeled Perchlorate ion ($\text{Cl}^{18}\text{O}_4^-$) was added to serve both as internal standard and correction for Perchlorate loss from sample preparation. The correlation coefficient of 0.9985 (Perchlorate ion 83) and 0.9989 and

(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. Initial Calibration Verification and Continuing Calibration

Initial calibration was verified by a second source standard (ICV) on 09-26-23. Percent recoveries were within required method limits (85-115% of the true value).

Continuing calibration standards were analyzed at 10-injections interval. A total of six daily standards were carried out on 10-09-23 and 10-10-23, 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. Perchlorate was spiked and reported for LCS/LCSD and MS/MSD. Percent recoveries (%R) were within the established QC limits. Raw data for both un-spiked sample and spiked QC samples were matching the reported values.

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

4.10.5. Field duplicate sample and its associated sample: Field sample FDUP01-102023 was identified as field duplicate of sample TMW16102023. No Perchlorate was detected in the field sample or associated field duplicate sample.

4.10.6. Raw data was submitted for all samples. Sample BGMW12102023 (Lab ID#23J027-05) was reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

4.11. 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 10-02-23. Samples were prepared (digested) for both total and dissolved metals on 10-19-23. Samples and QC samples were analyzed on 10-20-23 for total and dissolved metals by ICP MS. Water samples were preserved and filtered in the lab for dissolved metals analysis. Therefore, two sets of data were generated, one for total metals and one for dissolved metals.

4.11.2. Initial Calibration and Continuing calibration standards

Initial calibration was performed at the start of each analysis day on 10-20-23. Tune performance report was generated at the start of each analysis day, before initial calibration. It was within method's acceptance criteria. Initial instrument (ICP-MS) calibration for this method was acceptable. Five levels of concentration were used for each initial calibration. One method blank and one calibration standard was used for each daily check standard. A range of concentrations of standards was used for calibration. The concentrations used are summarized as follow:

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

Initial and continuing calibration verification standards for each element was within acceptable limit of 90-110 percent of the true value. Continuing calibration standards were analyzed at the frequency required by the method. Results for the ICP Interference Check Solutions (ICS-A and ICS-AB) were within the control limits of $\pm 20\%$ 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 MW23102023 was selected to be spiked as MS/MSD. Recoveries of LCS/LCSD and MS/MSD were all within the acceptance limit of

80-120% for both total and dissolved metals. However, few metals failed the acceptable QC limits in MS/MSD as shown in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	MW23102023 (Lab ID#J027-01) MS%	MW23102023 (Lab ID#J027-01) MSD%	QC Limit %	MW23102023 (Lab ID#J027-01) MS%	MW23102023 (Lab ID#J027-01) MSD%	QC Limit %
Aluminum	√	√	84-117	√	√	84-117
Antimony	√	√	85-117	√	√	85-117
Arsenic	√	√	84-116	√	√	84-116
Barium	113	83*	86-114	43*	47*	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	333*	67*	85-117	867*	-33*	85-117
Thallium	√	√	82-116	√	√	82-116
Vanadium	√	√	86-115	√	√	86-115
Zinc	√	√	83-119	√	√	83-119

*Outside control limits

Therefore, the positive results in parent sample will be qualified as estimated value “J”.

Results for method blanks were acceptable and no contamination was found in the method blanks. Calibration blanks were analyzed after each continuing calibration standard. Sample MW23102023 was also used for serial dilution for both total and dissolved metals. The serial dilution analysis (at 5-fold dilution) was within 10% difference of the initial analysis. The same sample was used for spike addition (analytical spike). All results were within the QC limit of (80-120%) for total and dissolved metals.

4.11.4. Field duplicate sample and its associated sample: Field sample FDUP01-102023 was identified as field duplicate of sample TMW16102023. Results and %RPD for field sample and associated field duplicate sample are listed in the table below:

ANALYTE	Total Metals			Dissolved Metals		
	TMW16102023 (Lab ID# J027-03) µg/L	FDUP01-102023 (Lab ID# J027-04) µg/L	% RPD	TMW16102023 (Lab ID# J027-03) µg/L	FDUP01-102023 (Lab ID# J027-04) µg/L	% RPD
Aluminum	400J	430J	6.65	U	U	--
Antimony	U	U	--	U	U	--
Arsenic	U	U	--	U	0.63J	200
Barium	20	24	18.2	12	13	8.0
Beryllium	U	U	--	U	U	--
Cadmium	U	U	--	U	U	--
Calcium	4300	4300	<1	4300	4300	<1
Chromium	12	12	<1	1.4J	2.1J	40.0
Cobalt	U	U	--	U	U	--
Copper	6.0	5.4J	10.5	U	2.9J	200
Iron	210J	240J	13.3	U	U	--
Lead	0.91J	0.77J	16.7	U	U	--
Magnesium	580	580	<1	450J	430J	4.54
Manganese	10	12	18.2	3.4J	8.2	82.8
Nickel	9.1	9.2	1.09	8.7	7.4	16.1
Potassium	650J	660J	1.53	580J	590J	1.71
Selenium	U	U	--	U	U	--
Silver	U	U	--	U	U	--
Sodium	470000	450000	4.53	480000	460000	4.25
Thallium	U	U	--	U	U	--
Vanadium	8.2	8.6	4.76	7.8	8.7	10.9
Zinc	U	U	--	U	U	--

4.11.5. Raw data was submitted for all samples. Sample BGMW12102023 (Lab ID#23J027-05) was reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

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

4.12.1. Technical Holding Times

Holding time from sample collection to extraction and extraction to analysis was met for twelve (12) water samples requested for this method. Water samples were collected on 10-02-23. Samples were prepared (digested) on 10-19-23 for Mercury and 10-21-23 for dissolved Mercury. Samples were analyzed on 10-19-23 for Mercury and on 10-21-23 for dissolved Mercury. All 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 10-19-23 and 10-21-23. The correlation coefficient of at least 0.999902 and 0.999968 was calculated to show the linearity of each calibration curve. The concentration of standards used for calibration ranged from 0.2 – 5.0 µg/L.

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

4.12.3. Quality Control: Data for all the QC samples were within acceptable control limits. The QC samples consisted of one method blank and one set of LCS/LCSD for Mercury and dissolved Mercury. Sample MW32102023 was selected by lab to be spiked as MS/MSD for dissolved Mercury only. Percent recoveries and % differences were within the control limits for LCS/LCSD and MS/MSD for both Mercury and dissolved Mercury. Sample MW32042023 was also used for serial dilution for dissolved Mercury. Method blank data was reviewed and no Mercury contamination was found in method blanks.

4.12.4. Field duplicate sample and its associated sample: Field sample FDUP01-102023 was identified as field duplicate of sample TMW16102023. No Mercury was detected in sample and associated field duplicate sample.

4.12.5. Raw data was submitted for all samples. Sample BGMW12102023 (Lab ID#23J027-05) was reviewed as stage 3 data deliverable. Raw data for this sample with the related QC samples was reviewed for stage 3 data validation. The results calculated from the raw data, agreed with all the results reported in data summary reports.

5.0 CONCLUSION

SDG #23J027 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.