

Mr. Alan York
Associate Director, Office of Water Quality
Division of Environmental Quality
Department of Energy and Environment
5301 Northshore Drive
North Little Rock, AR 72118-5317

Dear Mr. York:

We have completed our review of the latest Quality Assurance Project Plan (QAPP) for the Arkansas Department of Environmental Quality (ADEQ) Underground Injection Control (UIC) Program, #G-006221-22. I am pleased to inform you that the QAPP is approved for three years. This QAPP expires on August 18th, 2025.

Enclosed is one completed QAPP signature pages for your records. In any future correspondence relating to this QAPP, please reference Q-track #22-335. If you have any questions, please contact me at (214) 665-7313.

Sincerely,

Mike Vaughan /s/

Mike Vaughan, Project Officer
EPA Region 6
Community Infrastructure Section (WDAI)
Dallas TX

Enclosure

**QUALITY ASSURANCE PROJECT PLAN
ARKANSAS DEPARTMENT OF ENERGY AND ENVIRONMENT
ARKANSAS DIVISION OF ENVIRONMENTAL QUALITY
UNDERGROUND INJECTION CONTROL PROGRAM
FEDERAL FISCAL YEAR 2022-2023**



ARKANSAS
ENERGY & ENVIRONMENT

E & E UIC Coordinator

Linda A. Hanson

Date: June 8, 2022

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Date: 6/13/2022

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US EPA Project Officer

Michael Vaughan 08/08/2022

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Date: August 4, 2022

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US EPA Quality Assurance Officer

Date: _____

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Region VI



**UNDERGROUND INJECTION CONTROL
QUALITY ASSURANCE PROJECT PLAN
FOR
FLUID CHEMICAL ANALYSIS
DEPARTMENT OF ENERGY & ENVIRONMENT
DIVISION OF ENVIRONMENTAL QUALITY
Federal Fiscal Years 2023 - 2025**

Prepared by:

**Linda A. Hanson, M.Sc., P.G., UIC Coordinator
and
Lessie Redican, Branch Manager, Water Laboratory, Office of Water Quality**

June, 2022

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Element A3 - Distribution List

Copies of this document will be received by:

- Alan J. York, Associate Director, Office of Water Quality, DEQ
- Bryan Leamons, P.E., Senior Operations Manager, Office of Water Quality, DEQ
- Jamal Solaimanian, Ph.D., P. E., Engineer Supervisor, Permits Branch, Office of Water Quality, DEQ
- Linda A. Hanson, M.Sc., Geologist P.G., UIC Coordinator, No-Discharge Permits Section, Permits Branch, Office of Water Quality, DEQ
- Lessie Redican, Branch Manager, Water Laboratory, Office of Water Quality, DEQ
- Jonathan Westmoreland, Quality Control and Laboratory Accreditation Manager, Division of Energy
- Gary Looney, Assistant Director and UIC Coordinator, Arkansas Oil and Gas Commission, Division of Energy
- Michael Vaughan, EPA Project Officer, EPA Region 6, Community Infrastructure Section (WDAI).

Element A4 - Project / Task Organization

The Project

This project consists of semi-annual sampling of spent brine at Five (5) bromine chemical manufacturing facilities in Union and Columbia Counties during the Federal Fiscal Year (FFY) 2022. Three of these facilities are owned by LANXESS Corporation, and two are owned by Albemarle Corporation. These companies extract brine from the Smackover Formation at a depth of approximately 8,000 to 9,000 feet below ground surface. After the bromine has been extracted, the remaining brine (spent- or spent- brine), is re-injected into the Smackover Formation through Class V Underground Injection Control (UIC) wells. These brine production and debrominated-brine (spent brine) UIC disposal wells are regulated by the Arkansas Oil and Gas Commission (AOGC), which regulates all Class II UIC and Class V UIC-debrominated-brine disposal wells. The Arkansas Division of Environmental Quality (DEQ) regulates all Class I, Class III, Class IV, and all Class V disposal wells ***except for the Class V UIC debrominated-brine disposal wells.***

To demonstrate that the spent brine is simply the brine from which bromine has been extracted, and has not been contaminated with halogenated hydrocarbons from the various manufacturing processes, the AOGC agreed in a Memorandum of Understanding (MOU) for the Arkansas Division of Environmental Quality (DEQ) to sample and analyze the spent brine two times each year. Five (5) samples of spent brine are collected annually, five (5) in the spring and five (5) in the fall at each of the facilities by a DEQ Underground Injection Control (UIC) geologist or other qualified personnel. These samples are then analyzed for semi-volatile organic compounds (SVOCs) volatile organic compounds (VOCs), and metals by an Arkansas-accredited laboratory.

Department of E & E Staff

DEQ, Office of Water Quality (OWQ)

- Alan J. York., Associate Director, OWQ
- Bryan Leamons, P.E., Senior Operations Manager, OWQ
- Jamal Solaimanian, Ph.D., P.E., Engineer Supervisor, Permits Branch, OWQ
- Linda A. Hanson, Geologist M.Sc., P. G., UIC Coordinator, Permits Branch, OWQ
- Lessie Redican, Branch Manager, Water Laboratory, OWQ

OE, AOGC

- Gary Looney, Assistant Director, UIC Engineer Supervisor and Program Coordinator, AOGC, OE

OE, Shared Services

- Jonathan Westmoreland, Quality Control and Laboratory Accreditation Manager, OE

The structure of the ADE&E is documented on the Personnel Charts included in Appendix 1 of this document.

Sample collection of the injectate of the Class V UIC debrominated-brine disposal will be performed by DEQ UIC staff. Chemical analyses will be performed at the E&E laboratory or at a State of Arkansas-accredited contract laboratory. The results will be reported to the AOGC and to the appropriate bromine chemical manufacturing facility.

Element A5 - Problem Definition / Background

The specific purpose of this UIC Quality Assurance Project Plan (QAPP) is to protect underground sources of drinking water and provide quality chemical data concerning the volatile organic compounds, semi-volatile organic compounds, and metals content in the spent brine that is injected into the Class V UIC brine-disposal wells.

If it is determined that water quality violations may have occurred as a result of underground injection or that an underground source of drinking water is threatened, a sampling program relating to those concerns would be developed.

This QAPP is based on the ADP&EC Regulation 17, UIC regulations in 40 CFR 146-148, the Safe Drinking Water Act, 40 CFR regulations concerning environmental measurements, and the Memorandum of Understanding between the Arkansas Department of Pollution Control and Ecology (now DEQ) and the AOGC. The sampling and analytical procedures are adequate for use in enforcement.

Element A6 - Project / Task Description

Twice each year, five (5) spent brine samples are collected at five (5) bromine plants operated by LANXESS Corporation and Albemarle Corporation. At each site, at the last sampling point before the spent brine enters the pipeline to the Class V injection wells, two 40 mL glass vials with septa are filled without headspace for volatile organic analyses, a 1 liter amber glass jar is filled for semi-volatile organic analyses, and a 125 mL plastic bottle is partially filled for metal analyses. A trip blank (a 40 mL glass vial with septa) for the volatiles accompanies the containers. A duplicate set of samples is collected at one sampling location each day in three 40 mL glass vials with septa, a 1 liter amber glass jar for semi-volatile organic analyses, and a 125 mL plastic bottle for metal analyses. Two field blanks (40 mL glass vials with septa) are collected at every sampling point. The samples are then submitted to an accredited laboratory along with a chain-of custody form. A typical sample labeling procedure and a chain-of-custody form are in Appendices 3 and 4. The brine stream contains small air bubbles and is very hot, so the sample bottles are cooled slightly in ambient air before placing them on ice to prevent breakage of the glass sample containers.

In FFY1994 a Class V brine injection well sampling procedure was established. At that time the feed brine, spent brine, and leachate samples were collected from open ponds. Subsequently the ponds were replaced by tanks. Feed brine has not been sampled in recent years. On January 21, 2011, the Dow Chemical Company's Class I hazardous waste disposal UIC well began injecting the wastewater and leachate from the Albemarle West Plant. Since the leachate was no longer being disposed of in the Class V brine injection wells, sampling of leachate at these locations was discontinued effective Spring of 2011.

If volatile organic compounds are found to be present in those samples in concentrations above background levels for spent brine, then DEQ and AOGC UIC staff would proceed with further investigation into the possibility that unpermitted wastewaters are being improperly disposed into the Class V spent brine injection wells.

In the event evidence is found that existing treatment systems are being by-passed, the DEQ and AOGC would proceed with joint enforcement action against the operator.

Element A7 - Quality Objectives and Criteria for Measurement Data

The objectives of this QAPP are to provide data that is as complete as possible with the precision and accuracy necessary to support proper interpretation and decision making regarding regulatory compliance. The data must also be representative of the media and conditions being measured, and must be calculated and reported in units which allow comparison of data bases. The data is evaluated by statistical techniques for trends and is compared to water quality standards and criteria. Compliance data is compared to the permit limits to verify compliance.

The quality of the data should be sufficient to support regulatory actions. Errors resulting from faulty quality assurance could skew trend analyses and cause misappropriation of the DEQ's efforts for problem correction. Errors in compliance data could cause misdirected enforcement action.

The representativeness of the data will be assured by the proper selection of sampling sites and sampling methods. For example, a representative point to sample the injection fluid is at the wellhead.

In order to assure comparability among data from different sites or dates of collection, all values will be reported in milligrams per kilogram or milligrams per liter unless extremes of data render these units cumbersome. The overall precision of measurement is a mixture of sampling and analytical factors. Analytical precision is easier to control and quantify than sampling precision.

Goals for achieving our data quality objectives for sampling and analysis are precision, accuracy, and completeness.

Sampling precision will be determined by collecting and analyzing field duplicate samples. DEQ UIC staff will duplicate sampling conditions and sampling methods when collecting duplicate samples.

Precision of analytical data will be assessed using either method specific or laboratory established control limits. Duplicate sample results will be utilized to calculate the Relative Percent Difference (RPD). This RPD will be evaluated against laboratory established ~~upper and lower~~ control limits.

The accuracy of the laboratory analyses will be determined by calculating the percent recoveries of the analytes in Laboratory Control Samples (LCS). Percent recoveries will be evaluated against either method specified or laboratory-established upper and lower control limits.

Completeness is defined as the percentage of measurements made which are judged to be valid. The completeness goal is that a sufficient amount of valid data be generated. For this plan, it is believed that a completeness level of 100% should be achieved. Five (5) spent brine samples are collected twice each year, during two sampling events: spring and fall. Since plant compliance depends upon contaminant level results and due to the small number of samples, 100 % completeness is necessary and obtainable. In the event that any samples fail laboratory QC limits, a replacement sample would be collected.

Element A8 - Special Training Requirements / Certification

Hazardous Waste Operations and Emergency Response (HAZWOPER) training and certification requirements are necessary in order to complete the field tasks, which are usually done by a DEQ geologist rather than field technicians. Analyses will be performed by the Department of DEQ OWQ Laboratory or by an DEQ OWQ-contracted Arkansas-accredited laboratory. All laboratory analysts must have documented demonstrations of proficiency for each analysis.

Element A9 - Documentation and Records

The documentation for sampling will consist of sample number and type, time, location, and site operator written directly on the containers, on labels applied to the outside of the containers, or written directly on the plastic zip top bag containing the containers. This information is repeated on the chain-of-custody form. The documentation for chemical analyses will consist of the sampler writing the identifying information directly on the sample container with an indelible marker, the chain-of-custody form (see Appendix 4), raw data and records pertaining to the final report (reagents and standards preparation, sample preparation and calibrations), and final reports, which include Quality Control/Quality Assurance QA/QC results and corrective action reports. Any report generated by an Arkansas accredited contract lab has the responsibility to maintain all raw data and records pertaining to the report for at least three years. Copies of final reports will be sent to AOGC and will remain permanently in the bromine plant spent brine files at the DEQ Office of Water Quality.

Element B1 - Sampling Process Design (Experimental Design)

Five (5) samples of spent brine (the Smackover brine from which the bromine has been removed and replaced by chlorine) from five (5) facilities (three LANXESS Corporation plants and two Albemarle Corporation plants) are collected two times each year during sampling events, usually in the spring and in the fall. The sample points are determined by the layout of the manufacturing plants and the disposal wells. The samples will be collected at the last available sample point before the disposal wells, which generally is on or near the plant, before the flow-lines to the individual wells. The samples will be analyzed for volatile organic compounds, semi-volatile organic compounds, and metals by EPA Methods listed in this plan. All the measurements are classified as critical.

Element B2 - Sampling Methods Requirements

This sampling and the analyses are done by DEQ for the AOGC, which regulates the Class V UIC spent brine disposal wells that return the brine to the Smackover Formation (the source of the initially produced brine). In addition to the Class V well samples, there may be unscheduled samples collected from the injection line at the last available sampling point before disposal in the Class I wells, if warranted.

The samples will be collected utilizing the guidelines found in Section 3.0 of Chapter 1 and in Chapters 10 and 11 (including the documents hyperlinked in Chapter 11) of EPA's publication *Test Methods for Evaluating Solid Waste*, SW-846, available online at www.epa.gov/SW-846/main.htm and *Standard Methods for the Examination of Water and Wastewater* (as approved in 40 CFR 136).

The spent brine samples are taken from the injection line at the last available sampling point before disposal in the injection wells. Sample times and locations will be recorded.

Grab samples will be collected in 40 mL glass vials with septa (for volatile organic compounds), 1 liter amber glass jars (for semi-volatile organic compounds), and 125 mL plastic bottles to which HNO₃ was added at Laboratory Services (for metals). Sample containers are purchased from that certifies a level of cleanliness appropriate to the analytical method used. The containers will be used directly from their cartons with no further cleaning, and filled to the levels recommended by the laboratory. The containers will be disposed of properly after one use.

All samples will be received at the laboratory in a time frame that will allow analyses to be completed within holding times of each particular method.

Element B3 - Sample Handling and Custody Requirements

The purpose of the chain of custody procedure is to demonstrate the reliability of evidence by creating an accurate written record of the possession of the sample from collection to possible introduction into evidence. This procedure will also insure that the samples are collected, transferred, stored, analyzed, and disposed of properly only by authorized personnel.

Custody

A sample is in custody if it is in any one of the following states:

- A. In actual physical possession.
- B. In view, after being in physical possession.
- C. In physical possession and locked up.
- D. In a secure area, restricted to authorized personnel.

Sample collection

Sample containers are labeled by the sampler on site at the time of collection and indicate the station number and, the date and time of collection, the name of the collector, and any preservative used. The time and location are recorded on the chain of custody form (Appendix 4). The sample collector is responsible for the care and custody of the samples until they reach the laboratory. The sample collector must provide the proper storage conditions and insure the delivery of the samples within the permitted holding times. The samples must be in their physical possession or in their view or stored in a locked place at all times. The samples are kept on ice in coolers until they reach the laboratory. Samples must be accompanied by a chain-of-custody record form (Appendix 4) that includes the name of the investigation, the collector's signature, date, time, type of sample, number of containers, preservative used, condition of the samples, and analyses required.

Maximum Holding Times

Analysis	Maximum Holding Time
Trace Metals	6 months from collection to analysis
Semi-volatile Organics	7 days from collection to extraction; 40 days from extraction to analysis
Volatile Organics	7 days from collection to analysis

Element B4 - Analytical Methods Requirements

All analytical procedures used must be referenced in *Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act* 40 CFR, Part 136.

Metal Analysis

For total recoverable metals, water samples are analyzed using modifications of EPA 200.8, *Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry, Revision 5.4 (1994)* and EPA 200.7, *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma – Atomic Emission Spectrometry, Revision 4.4, (1994)*

The metals determined are:

Aluminum - total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4.(1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Arsenic - total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Barium - total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Beryllium - total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Boron - total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Cadmium- total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4, (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Calcium - total, mg/L, Inductively Coupled Plasma – Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Chromium- total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4 (1994);
OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Cobalt - total, mg/L, Inductively Coupled Plasma - Mass Spectrometry Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Copper - total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Iron - total, mg/L, Inductively Coupled Plasma – Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Lead - total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Magnesium - total, mg/L, Inductively Coupled Plasma – Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Manganese - total, mg/L, Inductively Coupled Plasma – Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Nickel - total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Potassium - total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Selenium- total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Sodium - total, mg/L, Inductively Coupled Plasma – Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Vanadium - total, mg/L, Inductively Coupled Plasma - Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Zinc- total, mg/ L, Inductively Coupled Plasma- Mass Spectrometry, Method 200.8, Rev. 5.4 (1994); OR by Inductively Coupled Plasma – AES, Method 200.7, Rev. 4.4 (1994)

Volatile Organic Compounds

Volatile organic compounds are analyzed by Gas Chromatography / Mass Spectrometry (GC/MS), Method 8260C, Rev. 3, August 2006; OR most recently approved revision at a local TNI-Accredited laboratory with an Arkansas Department of E & E contract.

Semi-volatile Organic Compounds

Semi-volatile organic compounds are analyzed by Gas Chromatography / Mass Spectrometry (GC/MS), Method 8270D, Rev. 4, January 1998; OR most recently approved revision at a local TNI-Accredited laboratory with an Arkansas Department of E & E contract.

Decontamination and Waste Disposal

No decontamination waste will be generated.

All waste solvents generated by extraction procedures are collected and recycled by a hazardous waste treatment facility.

Specific Performance Requirements

There are no specific performance requirements for the elements of this project.

Corrective Actions

Documentation

The analyst must document whenever a problem exists with the samples, sample data, or Quality Control data. The Corrective Action/Preventive Action (CAPA) Form will be used to document problems and corrective actions with the sample, (holding time errors, preservation errors) sample data, (outliers, matrix interference) or Quality Control data (out of control). The CAPA will be sent to the Water Laboratory Branch Manager or the Chemist Supervisor. The Water Laboratory Branch Manager or designee must sign off on any data that must be voided or coded.

Sampling

Problems with the samples will be documented on the Incident Report Form and the Laboratory Branch Manager or the Chemist Supervisor will contact the sampler. Corrective actions will include training and communicating sampling requirements to individuals charged with sample collection responsibility.

Analysis

QC Activity	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank	Response < Reporting Limit	Prepare another blank, determine cause
Initial Calibration	COV >0.995	Reanalyze standards, make new standards
QC check standard	Method or lab established control limits	Reanalyze standards, make new standards
Continuing Calibration	Method limits	Recalibrate and reanalyze samples
Sample Duplicates	Precision within Limits	Reanalyze, code results
Matrix Spike Duplicates	Precision within Limits	Reanalyze, determine cause, code results
Matrix Spike Duplicates	Recoveries within Limits	Reanalyze, determine cause, code results

Analytical Equipment (at contract laboratory).

Equipment	Parameter
Gas Chromatograph with a Mass Spectrometer Detector capable of meeting project and method requirements.	Volatile Organic Compounds
Gas Gas Chromatograph with a Mass Spectrometer Detector capable of meeting method requirements.	Semi-Volatile Organic Compounds
Inductively Coupled Plasma instrument with Mass Spectrometer or Optical Emission Spectrometer capable of meeting project and method requirements	Metals: Aluminum, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Nickel, Potassium, Selenium, Sodium, Vanadium, Zinc

Element B5 - Quality Control Requirements

Sampling

Field duplicate samples will be collected at a rate of 10%, or a minimum of one per sampling event. Each field sampler must collect a field duplicate consisting of four additional 40 mL glass vials with septa, two additional 1 liter amber glass jars, and two additional 125 mL plastic bottles at one site during the sampling event.

Laboratory Performance Checks

The performance of the laboratory will be checked with a scheduled system of duplicate samples, spiked samples, and check samples from an outside source.

- A. All chemical analyses possible will be checked for accuracy by the analysis of Laboratory Control Samples (LCS). A minimum of one LCS will be analyzed per batch of twenty samples. These spiked samples will be prepared by the addition of a known amount of target analyte(s) to an aliquot of de-ionized water, free of target analytes and organics. The results must be entered in the LIMS QC system and verified to be within the control limits.
- B. Laboratory Precision and the effect of the matrix on analyte recovery will be determined by the preparation and analysis of a minimum of one Matrix Spike/Matrix Spike duplicate (MS/MSD) per batch of twenty samples. Matrix Spike/Matrix Spike duplicate samples are prepared by adding a known amount of target analyte(s) to an aliquot of the sample which has a field duplicate. The precision and recoveries of the MS/MSD will be compared to method or lab generated acceptance criteria. The results must be entered in the LIMS QC system and verified to be within the control limits.
- C. Proficiency testing samples from The NELAC Institute (TNI) accredited PT providers will be analyzed semi-annually.. The analyst will perform the analysis without knowing the expected value.

Precision

The precision of the data will be determined from the field duplicate samples and the laboratory-spiked replicate samples.

The control limits for precision are either method specified or lab established.

Field Precision

The field precision will be based on the relative percent differences (RPD) between the sample (S1) and its field duplicate (S2). The control limits will be based on laboratory-established control limits. The RPD will be calculated as follows:

$$RPD = \{S2 - S1\} \div \{ (S2 + S1) \div 2 \} \times 100$$

Laboratory Precision

The values from laboratory-spiked (LS1 and LS2) replicate samples will be used to evaluate laboratory precision. The control limits will be based on method control limits or lab established limits. The RPD will be calculated:

$$RPD = \{LS2 - LS1\} \div \{ (LS2 + LS1) \div 2 \} \times 100$$

Accuracy

The control limits for accuracy are based on method or lab established limits and will be based upon the percent recovery of the laboratory control samples (LCS).

The percent recovery (P) is defined as:

$$P = 100 \times \{(\text{Final Concentration} - \text{Initial Concentration}) / \text{Spike added}\}$$

The analysis will be considered out of control if the recovery of the LCS is outside of the acceptance criteria for that parameter.

Corrective Action

The purpose of a corrective action is to document and promptly address major and/or minor problems, and to develop a plan that will eliminate the potential for repetition of the problem. Corrective actions are taken when:

- A. Quality Control checks reveal a problem
- B. The Quality Control data is out of control.
- C. Deficiencies are cited during an audit.
- D. Data is determined to be questionable by an outlier test.

Acceptance Criteria and Corrective Actions for Quality Control Checks

QC check	Acceptance Criteria	Corrective Actions
Reagent blanks	< Reporting limit	Verify reagent sources. Review preparation and storage procedures. Discard contaminated reagent. Document on incident report
Trip blanks	< Reporting limit	For common volatile contaminants (e.g. methylene chloride) the trip blanks are allowed to have 5X the reporting limit. The presence of any detected contaminant is reported.
Laboratory Control Sample (LCS)	% recovery within method specified or laboratory-established limits	Check spiking solution; re-prepare LCS and re-analyze LCS with associated samples.

Duplicates And Matrix Spikes

Duplicate Type	Corrective Action
Field Duplicates	Reanalyze samples if possible. If samples are still out of control, flag the data from sampling event. Document on incident report.
Matrix Spikes/Matrix Spike Duplicates	Reanalyze samples if possible. Check spike solution. Check for matrix interferences. Document on incident report. Qualify affected samples.

Acceptance Criteria and Corrective Actions for Performance Evaluation Samples

The PT providers will determine the acceptance criteria for performance evaluation samples. All results marked "not acceptable" will require corrective actions and written explanations to the QA Manager. Corrective actions will be:

- A. Checking calculations and data transcription.
- B. Checking calibration and calibration standards.
- C. Investigation of possibility of analyst error or improper technique.
- D. Investigation of possibility of instrument malfunction.
- E. Investigation of possibility of matrix interference.
- F. Documentation of corrective actions.

The analyst must document whenever a problem exists with the samples, sample data, or Quality Control data. The CAPA Form will be used to document problems with the sample (holding time errors, preservation errors), sample data (outliers, matrix interference), or QC data (out of control). The CAPA will be reviewed by the Branch Manager of the Office of Water Quality Laboratory, or the Chemist Supervisor. Any voided data must be approved by the Laboratory Branch Manager or designee.

Representativeness

The determination whether the results from a sample are truly representative of the brine are controlled by the sampling process. Site selection and training of the samplers will be the responsibility of the Project Manager. Representativeness of laboratory results will be assessed from the field duplicates. The control limits set for each field duplicate parameter are meant to assure proper sampling techniques.

Comparability

The laboratory must use two approaches to assure that data generated by the laboratory is comparable to data from other sources:

- A. Only EPA approved methods will be used on these projects.
- B. The laboratory will participate in two performance evaluation studies annually. These studies indicate the confidence of laboratory results and allow for comparison of the laboratory's results with other laboratories throughout the country.

Element B6 – Instrument/Equipment Testing, Inspection, and Maintenance Requirements

Inspections and Acceptance Testing of Instruments

All instruments purchased for this project will meet specific performance criteria before acceptance. The use of calibration standards will be used for these tests. Each instrument will be inspected during its scheduled cleaning and/or per manufacturer recommendations or operating instructions.

Final Acceptance

The final acceptance will be performed by the Chemist Supervisor or the Branch Manager of the Office of Water Quality Laboratory to assure compliance with purchase requirements.

Resolution of Deficiencies

If deficiencies are found during the testing procedure, the vendor will be given every opportunity to correct the problem within the available time allowed by the project and funding mechanisms.

Preventive Maintenance

A. Analytical Balance

- All analytical balances must be cleaned weekly and immediately after any chemical spills.
- The balance table must be kept neat and cleaned after any spills. Any spill that might interfere with trace analysis, such as mercury compounds, must be immediately and thoroughly cleaned.
- All analytical balances must be cleaned and checked by a balance service annually or whenever a problem is found.
- Daily Calibration checks are recorded before the balance is used.

B. Gas Chromatograph/Mass Spectrometer (at contract laboratory)

- The maintenance schedule should be followed.
- The following spare materials should be maintained on hand:
 1. filaments
 2. carrier gas (special order)
 3. septa
 4. vacuum pump oil
 5. molecular sieve

C. ICP/MS and ICP-OES (at contract laboratory)

- The maintenance schedule should be followed.
- The following spare materials should be maintained on hand:
 1. pump tubing
 2. inline filters
 3. nebulizer

D. De-ionized Water System

- Maintenance will be performed on the system by a vendor on a routine and scheduled basis.

Element B7 - Calibration Procedures

Calibration, the process of adjusting a piece of equipment to ensure it gives accurate answers, is one of the most important steps in any analysis. All laboratory equipment must be routinely calibrated. Calibration procedures are used on the following equipment.

7.1 Laboratory Instrumentation

Organics (at contract laboratory)

GC/MS as required by the method

Metals

ICP/MS as required by the method

ICP-OES as required by the method

7.2 Stock Standard Receipt and Traceability

7.2.1 Purchased Standards

Stock standards should be purchased from reputable vendors. They should be National Institute for Standards and Technology traceable and documented in the LIMS. They should be labeled with a lot number and an expiration date. Certificates of analysis should be filed for future reference, as part of the record.

7.2.2 Prepared Standards

Standards prepared from pure compounds are documented in the LIMS for traceability. This information should include: manufacturer, lot number, expiration date, weights and volumes taken, final concentration and preparer's initials.

7.2.3 Intermediate Standards and Spiking Solutions

The preparation of intermediate dilutions, mixed standards and spiking solutions must be recorded in the LIMS. The information recorded must include: lot number of the stock, concentration of the stock used, volume of the stock taken, final volume, final concentration of each component, preparer's initials and date.

7.2.4 Calibration Standards

Calibration standards are prepared from stock or intermediate standards. The preparation of calibration standards are documented in the LIMS. The information recorded must include: lot number of the stock, concentration of the stock used, volume of the stock taken, final volume, final concentration of each component, preparer's initials and date.

7.3 Instrument Calibration

All instruments and equipment will be calibrated according to the manufacturer's recommended procedures and the guidelines in the *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*, EPA-600/4-79-019.

7.3.1 General Lab Equipment

7.3.1.1 Analytical Balance, Sartorius Cubis Model MSA 124S-100-DI, or equivalent

Each day before use the balance must be checked for calibration by using a series of NIST traceable weights demonstrating the range of the balance (1g, 20g, 50g, and 100g). The balance reading for each weight must be within a range of acceptability for the balance to meet calibration check requirements. If any balance reading falls outside of the range of acceptability, the balance must be calibrated. The range of acceptability is calculated annually for each balance by calculating the average and standard deviation (SD) of a minimum of the last twenty readings of each weight. The acceptability limits are then defined as the average ± 3 SD.

7.3.2 Metals Laboratory

7.3.2.1 Thermo Scientific iCAP RQ ICP/MS and Agilent 5100 ICP-OES

1. Pre-calibration routine - The following pre-calibration routines must be completed prior to calibrating the instruments until it can be documented with periodic performance data that the instrument meets the criteria listed below:

For both instruments, initiate proper operating configuration of the instruments and data systems. Allow a period of not less than 30 minutes for the instruments to warm up.

For the ICP/MS, daily performance checks are analyzed following method requirements. If all criteria are not met, the analyst must re-tune the instrument and repeat checks until all criteria are achieved. For good performance adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. Adjust mass calibration if it has shifted by more than 0.1 amu from unit mass. Instrument stability must be demonstrated by running five (5) replicates of the tuning solution with resulting relative standard deviations of absolute signals for all analyses of less than 5%.

For the ICP-OES, a daily wavelength calibration solution is analyzed as a performance check before instrument calibration. If manufacturer specifications are not met, the analyst will perform corrective action and re-analyze the solution until the criteria are achieved.

Internal Standardization - Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. For full mass range scans, a minimum of three internal standards must be used. Procedures described in this method for general application detail the use of five (5) internal standards: scandium, yttrium, indium, terbium and bismuth. These were used to generate the precision and recovery data attached to this method. Internal standards must be present in all samples, standards and blanks at identical levels. This may be achieved by directly adding an aliquot of the internal standards to the CAL standard, blank or sample solution, or alternatively by mixing with the solution prior to nebulization using a second channel of the peristaltic pump and a mixing coil. The concentration of the internal standard should be sufficiently high that good precision is obtained in the measurement of the isotope used for data correction and to minimize the possibility of correction errors if the internal standard is naturally present in the sample. Depending on the sensitivity of the instrument, a concentration range of 20-200 $\mu\text{g/L}$ of

each internal standard is recommended. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

2. Calibration routine- Prior to initial calibration, set up proper instrument software routines for quantitative analysis. The instrument must be calibrated using one of the internal standard routines described above. The instrument must be calibrated for the analytes to be determined using the calibration blank and calibration standards prepared at one or more concentration levels. A minimum of three (3) replicate integrations are required for data acquisition. Use the average of the integrations for instrument calibration and data reporting.

The rinse blank should be used to flush the system between solution changes for blanks, standards and samples. Allow sufficient rinse time to remove traces of the previous sample. Solutions should be aspirated for thirty (30) seconds prior to the acquisition of data to allow equilibrium to be established.

7.3.3 Organics Laboratory (Contract Laboratory)

7.3.3.1 Gas Chromatograph/Mass Spectrometers are used for Volatile Organic Analyses and Semi-volatile Organics. The following guidelines are expected from the methods used.

The GC/MS will be tuned as recommended by the manufacturer. Once the proper ratios of the ions are obtained, this tuning will be checked using the appropriate amount of the tuning check compound. A background-corrected mass spectrum must meet all of the key M/Z criteria for the method being used. If all of the criteria are not met, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. These criteria must be demonstrated during each 12-hour shift.

Initial Calibration. A minimum of five (5) calibration standards must be prepared. The lowest concentration standard in the calibration curve must be at a concentration near or below the reporting limit. The others should correspond to the range expected in the samples but should not exceed the working range of the GC/MS system. Each standard should contain each analyte for the method. The response factors (RF) for each compound must have a percent relative standard deviation that meets the method requirements.

A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. The compounds system performance calibration checks (SPCCs) to be checked are listed in the method.

Daily Calibration. A mid-concentration standard containing each compound of interest, including all required surrogates, must be analyzed every twelve (12) hours. All target compounds of interest must be evaluated using a 20% variability criterion. If the percent difference or percent drift for a compound is $\leq 20\%$, then the initial calibration for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, some compounds will fail to meet the criteria. If the criterion is not met for more than 20% of the target compounds, then corrective action (i.e., re-analysis and/or re-prep of CCV, instrument maintenance, re-calibration) must be taken prior to the analysis of samples.

Element B8 - Inspection/Acceptance Requirements for Supplies and Consumables

- A. Supplies and consumables will be purchased by the Laboratory Branch Manager of the Office of Water Quality or the the contract laboratory. These persons are responsible for ordering the proper quality materials needed to support the project. The purchasing person will obtain the proper purchase order number from the E&E Fiscal Division and assure that the purchase is charged to the proper account/grant.
- B. The items ordered will be received by E&E Mail Room and/or Laboratory Services personnel or if from a contract laboratory, received by E&E Mail Room and/or Laboratory Services personnel or picked up from the contract laboratory by the person collecting the samples. The receiving person will inspect the material and check it against the packing slip. The packing slip will be turned in to the E&E Fiscal Division to ensure payment of any fees incurred.
- C. All chemicals and reagents will be dated and inspected for proper expiration date, then documented in the LIMS.
- D. The acceptance criteria for all Standards and Calibration materials will be traceability to the National Institute for Standards and Technology.

Below is a list of the supplies and consumables used for sampling:

Field Supplies

Metals: bottles and acid

Laboratory Supplies

Parameter standard, chemicals for spikes, dilution bottles

Metals

Reagents:	standards, acids, matrix modifiers
Glassware:	volumetric flasks, pipets
Other supplies:	pipet tips, pump tubes, test tubes, sample cups, graphite furnace tubes

Inorganic Nonmetallic Constituents

Reagents:	standards, color reagents
Glassware:	volumetric flasks, pipets, beakers, flasks, distillation flasks
Other supplies:	sample cups, pump tubes, computer paper, printer paper, electrodes

Individual Organic Compounds

Volatile Organics:

Reagents:	standards, methanol
Glassware:	volumetric flasks, pipets, syringes
Other supplies:	VOC vials

Extractable Base/Neutrals and Acids

Reagents:	standards, solvents
Glassware:	separatory funnels, volumetric flasks, pipets
Other supplies:	sample vials, syringes

Element B9 - Data Acquisition Requirements (Non-Direct Measurements)

No data will be collected or used from non-measurement sources.

Element B10 - Data Management

Field Data

The data collected in the field is recorded on the chain of custody form (Appendix 3). Upon receipt of the samples by the laboratory, the sample data, including date, time, and station number, is entered into the Laboratory Information Management System (LIMS) and issued a unique laboratory number.

Laboratory Data

Automated Methods: Data generated by automated procedures, e.g. ICP, GC/MS, is imported directly to the LIMS. All instrument-generated paper is stored according to DEQ's Records Retention Policy.

Control Mechanisms for Detecting/Correcting Error

The data is checked for errors at several points. The data must pass all precision and accuracy checks for both the field duplicates and the laboratory matrix spike replicates. The data must be within the allowed range, e.g. pH between 0 and 14 standard units. The data is manually checked for logical errors, e.g. a dissolved fraction that is greater than the total.

Data Handling Equipment and Procedures

The equipment used to handle the data servers and workstations as provided by DEQ Information Technology Services (ITS). Data will be processed; compiled and analyzed using purchased software including Excel spreadsheets, proprietary software used by the laboratory instruments or the LIMS.

Data Storage

The data in the LIMS is stored on a server at E&E and routinely backed up no less than once per day.

Data Use

The information that is generated is available to users from several sources. The data generated will be available to DEQ staff on the E&E computer system. The data also is provided to the Arkansas Oil and Gas Commission and to the respective plants. The Cadmus Group obtained spent brine injectate chemical characteristics information from the Arkansas Oil and Gas Commission for publication in the 1999 EPA publication EPA/816-R-99-014i: *The Class V Underground Injection Control Study, Spent Brine Return Flow Wells*, in order to obtain representative analyses of spent brine..

Element C1 - Assessments and Response Actions

The E&E Quality Assurance (QA) Manager will conduct an annual inspection of the laboratory in order to review and assess analytical procedures, laboratory personnel, facilities, instrumentation, laboratory quality control, and data handling. The QA Manager or designated personnel will also perform annual inspections of field duties to assess sampling methodologies, data handling, field quality control procedures, and personnel.

Laboratory performance will be checked using semi-annual Performance Testing (PT) Provider check samples. A sample for each measurement parameter will be analyzed and the results reviewed by the E&E QA Manager and Laboratory Branch Manager of the Office of Water Quality. The QA Manager or designee will conduct at least one field audit consisting of, but not limited to, sampling techniques, sample preservation, sample labeling, and field data handling techniques during the project period. A report on any deviations from the QAPP will be generated and distributed to the Project Manager, Branch Manager of the Office of Water Quality Laboratory, and Field Coordinator.

Corrective actions are taken when:

- A. quality control checks reveal a problem,
- B. the QC data is out of control,
- C. deficiencies are cited during an audit, or
- D. data is determined to be questionable by an outlier test.

If for any of the above reasons precision and/or accuracy data fall outside the boundaries of the control or acceptable recovery or bias standards, the analyst will consult his/her supervisor. If it is determined that the analytical system is out of control, the QA Manager, the Laboratory Branch Manager of the Office of Water Quality, or the Laboratory Chemist Supervisor is consulted and the system is brought back into control. At this time, all data sets containing precision or accuracy points that have shown the analysis to be out of control are re-analyzed, if possible

Element C2 - Reports to Management

A summary QA report will be prepared and submitted to the EPA Project Officer. The report will be prepared and submitted along with the final report. The report will include precision and accuracy data, an evaluation of the completeness of the data, and a discussion of any significant QA problems, as outlined below.

- A. QA management (any changes),
- B. Status of completion of the QAPP,
- C. Measures of data quality from the project,
- D. Significant quality problems, quality accomplishments, and status of corrective actions,
- E. Assessment of data quality in terms of precision, accuracy, completeness, representativeness and comparability, and/or
- F. QA report on field sampling techniques.

Quarterly progress reports will be generated by the Project Officer and distributed to EPA and the DEQ Office of Water Quality Associate Director and Program Manager.

Element D1 - Data Review, Validation, and Verification Requirements

Data Verification

The integrity of the data generated must be validated prior to entry into the database. The Laboratory Branch Manager of the Office of Water Quality and the Chemist Supervisor are responsible for ensuring that all of the laboratory data are properly reviewed and verified, and is in the proper format for submittal to the storage databases. The Project Manager is responsible for ensuring that all field and biological data are properly reviewed and verified, and is in the proper format for submittal to the storage databases. All the data produce must meet the data quality objectives outlined in Element A7. Data that does not meet the data quality objectives as outlined in Element A7 will not be input into the data storage databases.

Element D2 - Validation and Verification Methods

Data Validation

The integrity of the data generated will be verified at several points during the collection and reporting process. The principal point is the laboratory quality control check.

The laboratory control checks are described in Element B5. These checks consist of field duplicates and laboratory duplicates and spikes to monitor the levels of precision and accuracy of the collection and analytical processes.

The data processing checks are designed to assure the accurate transfer of the data from the laboratory report forms to the computer system. There are two points where the data is checked:

After the initial data entry, the data is manually checked by peer review from another analyst familiar with the method. The Laboratory Branch Manager of the Office of Water Quality and the Chemist Supervisor review the data reported for accuracy. The data is checked by the LIMS for holding time, recovery criteria, and other method requirements.

Outliers

Quality Control Data

Outliers from the laboratory quality control checks indicate sampling or analytical problems. All samples in these out-of-control situations will be re-analyzed or, if re-analysis is impossible, the data will be examined for any obvious causes.

Sample Data

When a value in a data set is suspiciously high or low, it must be examined to see if it must be discarded to avoid biasing the data set. The first check should be to see if there is any physical reason or any other explanation.

If a reason for the outlier value is not found, it must be tested to see if it is statistically judged to be an outlier. The suspect data point and the eleven (11) closest data points in the data set are used in the test.

Data Flow

Sample Collection:

The spent brine samples are collected by the DEQ Underground Injection Control geologist or other water quality sampling-trained personnel. All field data is reported to the lab on the chain of custody form. The samples are collected two times each year at a minimum at the same five (5) sampling points installed on the spent brine pipelines.

Laboratory:

- A. The spent brine samples received in the laboratory will follow the chain of custody procedure described in Elements A9 and B3. All of the field data is transferred to the bench sheets where analytical data is recorded.
- B. Precision and accuracy of data checked and results are recorded on quality assurance charts.
- C. Data is reviewed for completeness and mathematical errors and prepared for data processing.

Data Processing:

- A. Data is entered into a computer file. A printout of entered data is obtained and manually checked against laboratory forms.
- B. Data is scanned for out-of-range values.

Element D3 - Reconciliation with Data Quality Objectives

The results obtained from the spent brine sampling are evaluated twice each year when the chemical analyses become available. The precision and accuracy of the data developed for the project will be reported to the decision makers to show the limits that should be placed on the data.

All data generated by this project that meets the QA/QC requirements set forth by this QAPP will be used to establish trends and to indicate any variation from the trend that might indicate that plant process wastewaters or waters other than the spent brine are being injected into the Class V wells.

Copies of the analytical results are kept by the Laboratory Branch Manager of the Office of Water Quality, in the Underground Injection Control files in the Office of Water Quality No-Discharge Section Underground Injection Control Program files, and by the Arkansas Oil and Gas Commission.

APPENDIX 1

Organizational Charts for AR Dept. of Energy and Environment

At the time this document was submitted, the organizational charts for the Arkansas Department of Energy and Environment and its subsidiaries was not complete. Once these charts are completed and available, a copy or a link to the organizational charts for the following will be submitted to the Region VI EPA UIC Program:

Organizational Chart for the Arkansas Department of Energy and Environment:

Organizational Chart for the Division of Environmental Quality

Organizational Chart for the Office of Water Quality

APPENDIX 2
Sample Labeling Procedures

Typical Sample Label

DEQ personnel write the following sample information directly on the 1 liter amber bottles used for semi-volatile organic compound analyses.

- Date
- Sample identification number
- Number and sequence (i e. 2 of 3)

DEQ personnel write the following sample information directly on the 125 mL plastic bottle used for metals analysis with a permanent marker:

- Date
- Sample identification number
- Number and sequence (i e. 2 of 3)

DEQ personnel write the following sample information directly on the plastic zip top bag used to contain the 40 mL volatile organic compound vials:

- Date
- Sample identification number
- Number and sequence (i. e. 2 of 3)

APPENDIX 3

Chain-of-Custody Forms

For Compliance, Enforcement, or Emergency Samples

[illegible]Revision 1
October 1, 2019

ARKANSAS DIVISION OF ENVIRONMENTAL QUALITY CHAIN-OF-CUSTODY					
Date		Sampler (print)			
Site Identification					
Site Address					
Sample ID	Sample Remarks	Time (hhmm)	Latitude	Longitude	Lab ID
Lab Use Only: Custody seal on each container?: YES NO Labels/COC agree?: YES NO					
Date/Time	Relinquished By		Received By		
Date	Name/Title		Name/Title		
Time	Signature		Signature		
Date	Name/Title		Name/Title		
Time	Signature		Signature		
Date	Name/Title		Name/Title		
Time	Signature		Signature		
Date	Name/Title		Name/Title		
Time	Signature		Signature		