ROUTING AND TRANSMITTAL SLIP

Date 05/01/14

TO: *(Name, office symbol, room number, building, Agency/Post)*	Initials	Date
1. Blake Huff, Tribal Program Manager	BH	5/1/14
2. Tricia Pfeiffer, Water Quality Lead	TP	5/2/14
3. Karl Hermann		
4. Jeff Pritt, QA Reviewer	JP	5/2/14
5. Linda Himmelbauer, RQAM	LH	5/2/14
6.		
7.		
8.		
9.		
10.		

☐ Action	☐ File	☐ Note and Return
☒ Approval	☐ For Clearance	☐ Per Conversation
☐ As Requested	☐ For Correction	☐ Prepare Reply
☐ Circulate	☐ For Your Information	☐ See Me
☐ Comment	☐ Investigate	☐ Signature
☐ Coordination	☐ Justify	

REMARKS

QAPP
for the
Ambient Monitoring of Surfance Water and Groundwater Quality
on the
Fort Berthold Indian Reservation, North Dakota

Return to Blake Huff, 8P-TA

Tricia printed + added Appedix attachments

DO NOT use this form as a RECORD of approvals, concurrences, disposals, clearances, and similar actions

FROM: *(Name, org. symbol, Agency/Post)* Blake Huff, 8P-TA	Room No. – Bldg. 7227 Phone No. (303) 312-6718

OPTIONAL FORM 41 (Rev. 1-94)
Prescribed by GSA

QUALITY ASSURANCE PROJECT PLAN

for the

AMBIENT MONITORING OF SURFACE WATER AND GROUNDWATER QUALITY

on the

FORT BERTHOLD INDIAN RESERVATION, NORTH DAKOTA

CWA SECTION 106
THREE AFFILIATED TRIBES
NEW TOWN, NORTH DAKOTA

Describing Operations for the Years 2014-2019

Prepared for:

USEPA Region 8
1595 Wynkoop
Denver, CO 80202-2466

Prepared by:

Dana Price
TAT Project Manager
Three Affiliated Tribes
Environmental Division
404 Frontage Road
New Town, ND 58763

February 2014

A1 Title and Approval Page

QUALITY ASSURANCE PROJECT PLAN
for the
AMBIENT MONITORING OF SURFACE-WATER AND GROUNDWATER QUALITY
on the
FORT BERTHOLD INDIAN RESERVATION, NORTH DAKOTA

Describing Operations for the Years 2014-2019

Approval Signatures:

Dana Price ____________________ Date: 4-30-14
Dana Price, 106 TAT Project Manager
TAT Project Manager, Three Affiliated Tribes

____________________ Date: 4-30-14
Edmund Baker, Environmental Tribal Director
TAT QA Project Officer, Three Affiliated Tribes

Tricia Pfeiffer ____________________ Date: 5/2/14
Tricia Pfeiffer, Project Manager
Watershed and Aquifer Protection Unit, EPA Region 8

____________________ Date: ____________
Karl Hermann, EPA Project QA Officer
Water Quality Unit, EPA Region 8

____________________ Date: 5/2/14
Blake Huff, EPA Project Officer,
Tribal Assistance Program, EPA Region 8

____________________ Date: 5/2/14
Linda Himmelbauer, Director
Quality Assurance Program, EPA Region 8

A2 Table of Contents

A3 Distribution List

Name: Dana Price
Title: 106 TAT Project Manager; TAT Project Manager
Organization: Three Affiliated Tribes Environmental Division
Contact Information 204 W Main Street; New Town, ND 58763; 701-627-4569
dprice@mhanation.com

Name: Edmund Baker
Title: Environmental Tribal Director; Project Quality Assurance Officer
Organization: Three Affiliated Tribes Environmental Division
Contact Information 204 W Main Street; New Town, ND 58763; 701-627-4569
EBaker@mhanation.com

Name: Blake Huff
Title: Tribal Project Manager
Organization: US EPA Region 8
Contact Information: 1595 Wynkoop Street; Denver, CO 80202303- 312-6718
Huff.Blake@epa.gov

Name: Tony Ranalli
Title: USGS Water-Quality Data Management Lead
Organization: USGS Colorado Water Science Center
Contact Information PO Box 25046, MS 415 Denver, Co 80225; 303-236-6915
tranalli@usgs.gov

Name: Joel Galloway
Title: USGS Project Manager
Organization: USGS North Dakota Water Science Center
Contact Information 821 East Interstate Avenue; Bismarck, ND; 701-250-7419
jgallowa@usgs.gov

Name: Tricia Pfeiffer
Title: EPA Project Manager; TAT Water Quality Lead
Organization: Watershed and Aquifer Protection Unit, EPA Region 8
Contact Information: 1595 Wynkoop Street, 8EPR-EP; Denver, CO 80202; 303-312-6271
pfeiffer.tricia@epa.gov

Name: Karl Hermann
Title: EPA Project QA Officer
Organization: Water Quality Unit, EPA Region 8
Contact Information 1595 Wynkoop Street, 8EPR-EP; Denver, CO 80202; 303-312-6628
hermann.karl@epa.gov

Name: Jack Sheets
Title: EPA Lab Project Manager
Organization: US EPA Region 8 Laboratory
Contact Information: 16194 W. 45th Drive, 8TMS-L; Golden, CO 80403; 303-312-7793
schroeder.william@epa.gov

Name: John Hargraves
Title: Omaha District Water Quality Section Biologist
Organization: US Army Corps of Engineers
Contact Information: 1616 Capitol Ave; Omaha, NE 68102; 402-995-2347
john.g.hargrave@usace.army.mil

A4 Project Organization

A4.1 Tribal and USGS Roles and Responsibilities

Dana Price, Water Quality (106) Coordinator and TAT Project Manager, will be on the water sampling staff and will assist with sampling activities and site access. She will coordinate TAT Environmental Division support for the Project and establish the project schedule. She will coordinate with the USGS Project Manager in the data collection and interpretation of the study. The TAT Project Manager has the responsibility for preparing, maintaining, and implementing the QAPP for this project and managing day-to –day project activities. She will present the results and findings of the project to the TAT Project Quality Assurance Officer and EPA. She is responsible for approving the QA Project Plan for the Three Affiliated Tribes.

Edmund Baker, Tribal TAT Project Quality Assurance Officer and TAT Project Quality Assurance Officer, will be responsible for oversight of the TAT project manager. He is responsible for approving the QA Project Plan for the Three Affiliated Tribes. He will ensure that the QA Program is implemented, resolve any QA issues, and approve the TAT project reports. He is independent of all sampling activities.

Joel Galloway, USGS Project Manager, will be responsible for directing water-quality sampling tasks and objectives, sample collection, overall field activities, and provide oversight on the management and quality assurance of water-quality data. Quality assured data will be provided to the Water-Quality Data Management Lead and TAT Water-Quality Coordinator for entry into the USEPA AQWMS database and for interpretation. The USGS project lead will also coordinate with the Water-Quality Data Management Lead in the interpretation of data described in this QAPP. He will report project status/issues to both the TAT Project Manager and EPA Project Manager.

Tony Ranalli, USGS Water-Quality Data Management Lead, will be responsible for providing training to the TAT Water-Quality Coordinator in data quality assurance/control and data interpretation. He will assist the TAT Water-Quality Coordinator (Dana Price) with quality assuring and interpreting all data collected for TAT and ensuring all data collected are provided electronically and in reports to Three Affiliated Tribes. In addition, he will assist in the interpretation of data described in this QAPP as well as data collected in Lake Sakakawea by the North Dakota Department of Health and the U.S. Army Corps of Engineers.

A4.2 EPA Roles and Responsibilities

Tricia Pfeiffer, EPA Project Manager, reports to both the EPA Project QA Officer and the Water Quality Unit Chief. She is the responsible official for the Project and will coordinate the project schedule with the EPA Lab. The EPA Project Manager will keep the Water Quality Unit Chief informed on work accomplished in this plan and will assist with any problems or deviations that need to be resolved. The EPA Project Manager has the responsibility for the coordination of laboratory services. The EPA Project Manager will maintain the Project File with key documents. She is responsible for approving this TAT QA Project Plan

Karl Hermann, EPA Project QA Officer, reports to the Water Quality Unit Chief. The EPA Project QA Officer will approve the QA Project Plan and ensure that the QA program is implemented and assist with resolving any QA issues. He is independent of all sampling activities.

Blake Huff, Tribal Project Manager, reports to the EPA Tribal Assistant Program Manager. He is the EPA 106 grant project officer. He is responsible for approving this TAT QA Project Plan.

EPA Region 8 Laboratory will provide analytical services and technical support to the project as described herein following the current Region 8 Laboratory Quality Assurance Manual (**Appendix VI**). **Jack Sheets** will serve as the Region 8 Laboratory contact for the Project. **William Batschelet** is the Region 8 Laboratory QA Officer.

A5 Problem Definition/Background

The Fort Laramie Treat of 1851 originally established the Fort Berthold Indian Reservation, located in northwestern North Dakota (**Figure 1**). The Reservation occupies portions of six counties - Mountrail, Ward, McLean, Dunn, McKenzie and Mercer. The current boundaries as determined by an Act of Congress in 1891 encompass approximately 986,000 acres of which about one-half is held in trust by the United States for either the Three Affiliated Tribes or individual Native Americans.

The large, man-made Lake Sakakawea occupies 155,000 acres of land in the center of the Reservation. Created by the construction of the Garrison Dam on the Missouri River between 1947 and 1953, the lake stretches 178 miles in length between Williston and Riverdale, North Dakota with a drainage area of 181,400 square miles. The dam created a lake with a surface area at full pool of 368,000 acres encompassed by 1,300 miles of shoreline, six hundred miles of which lie within Reservation boundaries. Only one bridge spans Lake Sakakawea within the Reservation boundaries and it is located west of New Town at the Reservation's northern edge.

Lake Sakakawea provides municipal water for three of the five Reservation communities - Mandaree, White Shield and Twin Buttes, as well as the Four Bears community located directly across the river from New Town. The lake is also a major source of recreational opportunities including fishing, boating and water skiing. Industrial use of the lake resource is continuing to increase as energy development on the Reservation increases.

Other water resources on the Fort Berthold Indian Reservation include ephemeral streams, wetlands, small man-made impoundments that are used for livestock watering and wildlife habitat, and groundwater. Major streams on the Reservation are Bear Den Creek, Shell Creek, East Fork Shell Creek, Deepwater Creek, Moccasin Creek, and Squaw Creek. Groundwater occurs within glacial deposits and bedrock aquifers of the Fort Union Formation (Dingman and Grodon, 1954). All of the major streams flow into Lake Sakakawea and Dingman and Gordon (1954) state that the general movement of groundwater in aquifers that lie above river level is toward the Missouri River. Beneficial uses of Reservation waters include: 1) municipal domestic water supply, 2) recreation, 3) aquatic life, and 4) agricultural uses, including irrigation.

Land use consists of energy development, agriculture, and cattle grazing. The Fort Berthold Indian Reservation lies within the Williston Basin in eastern Montana and western North Dakota, which is one of the most intensive areas of energy development in the United States. On the Fort Berthold Indian Reservation in western North Dakota there are currently 1,087 active oil wells throughout the Reservation with hydraulic fracturing (fracking) occurring at most if not all wells. Most of the energy development on the Fort Berthold Indian Reservation has taken place only within the last 5 years and the number of oil wells on the Fort Berthold Indian Reservation is projected to increase by 1,700 in the foreseeable future (Tricia Pfeiffer, United States Environmental Protection Agency, personnel communication, 2012). Agricultural land use is predominant on the east side of the reservation and consists primarily of cereal crops with a small amount of row crops. Crops grown include wheat, barley, oats, corn, sunflower, alfalfa, and hay. Rangeland used for the cattle grazing is the predominant land use on the west side of the reservation.

The primary goal of the TAT Environmental Department is the protection of the water-quality of Lake Sakakawea because of its importance to the residents of the Fort Berthold Reservation as a domestic water source and for the recreational opportunities it provides. The varied land uses on the Fort Berthold Indian Reservation have the potential to influence the water quality of the streams and groundwater on the Reservation, and since these waters flow into Lake Sakakawea, potentially the lake as well. As a result, the TAT Environmental Department has identified the need for ambient water quality monitoring of surface-water and groundwater throughout the Reservation. Therefore, the six streams, selected groundwater wells, and springs will be sampled in the ambient water-quality monitoring program. Water-quality sampling of Lake Sakakawea will be done as part of this program. Water-quality data has been and continues to be collected from Lake Sakakawea by the North Dakota Department of Health (NDDH) and the U.S. Army Corps of Engineers (USACE) (NDDH, 2009; NDDH 2012, USACE, 2012). The R8 EPA Laboratory will send the USACE the appropriate sampling materials. The USACE will collect samples for this study at their current sampling locations highlighted in yellow on the table (see Appendix VII). Sample collection will follow the procedures of the QAPP, including sending the samples to the R8 Laboratory for analysis. The rest of the analysis process will be followed as described in the QAPP.

A6 Project/Task Description and Schedule

The goal of this ambient monitoring program is to determine the effects of land use on stream and groundwater quality on the Fort Berthold Indian Reservation. A secondary goal of the TAT Environmental Department is to continue water quality monitoring, so that potential long-term changes in water-quality over time can be detected.

The responsibility of the data collection will be shared between the TAT Environmental Department and the USGS. The USGS will provide training to the TAT Environmental Department in sample collection and processing, laboratory submittal procedures, and quality control evaluation and interpretation of the water quality data. The TAT Environmental Department will have responsibility for preparing the USEPA required yearly data assessment reports. The USGS will provide data interpretation and a published report in 2016.

Project Descriptions and Definitions

Samples will be collected and analyzed following the procedures described in section **B. Data Generation and Acquisition**. Data will be validated and reported by the EPA Region 8 Laboratory as described in the Laboratory QA Manual (**Appendix I**).

Purpose of Surface Water Sampling: Surface water sampling allows for the characterization and assessment of water quality conditions in relation to contaminant sources. The data allows for responsive public and environmental health decisions to be made.

pH (Potential for Hydrogen Ion) is a measure of the acidity or alkalinity of a solution. Neutral water has a pH of 7 at 25°C (77° F), the pH of alkaline water is >7, and the pH of acidic Water is <7.

Temperature is given is degrees Celsius (^{o}C) which can be converted to degrees Fahrenheit (^{o}F):

$^{o}F= 1.8 \times {}^{o}C + 32$

Dissolved Oxygen is necessary for good water quality. Natural stream purification processes require adequate oxygen levels in order to provide for life forms. As dissolved oxygen levels in water drop below 5.0 mg/l, aquatic life is put under stress (2.0-1.0 mg/L causes fish kill).

Conductivity is a measure of the ability of water to pass an electrical current. Conductivity in water is affected by the presence of inorganic dissolved solids such as chloride, nitrate, sulfate, and phosphate anions (ions that carry a negative charge) or sodium, magnesium, calcium, iron, and aluminum cations (ions that carry a positive charge). Organic compounds like oil, phenol, alcohol, and sugar do not conduct electrical current very well and therefore have a low conductivity when in water. Conductivity is also affected by temperature: the warmer the water, the higher the conductivity. For this reason, conductivity is reported as conductivity at 25 degrees Celsius (25 C).

Collection of the grab samples is critical for the project. The field measurements are important for completeness of the project and shall be collected with each water quality sample.

Project Schedule

Sampling will take place on four events per year. The specific dates for the sampling events, including travel on the days at both ends, are:

April Sampling Event

Monday, 4/7/2014 – Friday, 4/11/2014

26 Baseline Sites- All sites listed in **Table 1 and Table 2**

June Sampling Event

Monday, 6/9/2014 – Friday, 6/13/2014

24 Targeted Variable Sites-Variable Targeted Sites are to be determined. The 24 variable targeted sites have not been determined but the criteria on selection consist of choosing locations from existing areas known to have spills in close proximity to drainages that flow into Lake Sakakawea. Some of the locations will be selected based on close proximity to public water system intakes.

August Sampling Event

Monday, 8/11/2014 – Friday, 8/15/2014

24 Targeted Variable Sites-Variable Targeted Sites are to be determined by method mentioned above.

October Sampling Event

Monday, 10/13/2014 – Friday, 10/17/2014

26 Baseline Sites- All sites listed in **Table 1 and Table 2**

The project criteria for the variable targeted sites include existing areas known to have spills in close proximity to drainages that flow into Lake Sakakawea. Some of the locations will be selected based on close proximity to public water system intakes.

The tribe and USGS will receive the raw data from the EPA Lab for QA/QC review. The EPA Project Manager will review the data and then will send it to the TAT Project Manager. After the QA/QC review the TAT project manager will submit the data to the EPA project manager. The annual EPA required data assessment report for the previous year's data will be due by July 1st of the following year.

Objective 1: Collect water quality and hydrologic data needed to monitor the ambient water-quality conditions in the streams and groundwater of the Fort Berthold Indian Reservation, and to continue monitoring over the long-term so that potential changes in water quality over time can be detected.

Objective 2: Ensure that all data collected are available and reported to the USEPA and the public.

Task 1: Surface Water Sampling

Drainage Sampling

For baseline sampling, the six major creeks that drain into Lake Sakakawea being sampled are listed in **Table 1** and shown in **Figure 2**. Water-quality samples from the Bear Den Creek, Squaw Creek, Moccasin Creek, Shell Creek, East Fork Shell Creek, and Deepwater Creek locations will be analyzed for the full suite of compounds listed in **Table 3**. These locations will be sampled during the April (2 samples), May, June, August and October sampling events. This sampling design will aid in measuring trends and loads (Galloway and others, 2012). Streamflow also will be measured in each stream every time a water quality sample is collected. Streamflow data are necessary because potential changes in water-quality will be assessed by performing trends analysis on stream concentrations and loads. Many constituents have a well-defined relation with streamflow. Therefore, to account for changes in concentration over time the influence of streamflow on concentration needs to be removed before trends analysis on the concentration data can be performed.

For variable targeted sampling, data that will be considered consists of locations of existing oil and gas wells, records of related spills, locations of public water system intakes, locations of sampling by other entities (USGS), and perennial streams. Spill locations will be identified form the following website http://www.gagecartographics.com/spills. The variable targeted locations for FY 2014 have been identified and are listed in **Table 2** and shown in **Figure 2**. Some of these locations may become fixed while others will be replaced with a new locations dependent on land use activities that are identified in the future. Water-quality samples from these drainages will be analyzed for the full suite of compounds listed in **Table 4**.

Spring Sampling
Water-quality samples will be collected from springs during each sampling event and will be analyzed for the full suite of compounds listed in **Table 3**. The flow of each spring (discharge) will also be measured each time a water-quality sample is collected. The springs to be sampled will be selected during the first sampling event in April 2014. A field reconnaissance is needed to accurately assess the proximity of the springs to land use activities. The QAPP will be updated with the spring locations after the field reconnaissance. The springs will be sampled to determine the influence of spring discharge on stream water-quality and because they may function as "early warning indicators" of land use impacts and because they are in a shallow groundwater flow path. The shallow groundwater flow path results from the fact that the springs on the Fort Berthold Indian Reservation occur within the Sentinel Butte Formation and originate in lignite beds, which are usually at a shallow depth. We anticipate sampling 5 springs during the sampling events, but will be determined after reconnaissance. The QAPP will be amended in spring 2014 with the locations. If locations are not accessible, an alternative will be chosen and the QAPP will be amended.

Task 2: Groundwater Sampling

Water-quality samples will be collected from groundwater wells by USGS personnel and the TAT Environmental Department and will be analyzed for the full suite of compounds listed in **Table 3** twice each year, once in April and once in August at the same time the streams are sampled. Groundwater levels also will be measured each time a water-quality sample is collected. It is not possible to measure groundwater levels in domestic wells because they are sampled at the tap and there is no access to the well to be able to measure groundwater levels.

There are two objectives for sampling groundwater wells. One objective is to determine the influence of groundwater on the water-quality of each stream. It is important, especially given the ephemeral nature of these streams, to determine the degree to which groundwater influences the quantity and quality of surface water throughout the year. For example, if the constituents associated with the various land uses are introduced into groundwater will the contaminated groundwater discharge into streams throughout the year, only at certain times during the year, or will it bypass the streams altogether? A second objective is to obtain a representative sampling of groundwater quality in the various aquifers and land uses on the FBIR. The criteria for selecting groundwater wells were based on land use (we want to sample groundwater wells in agricultural areas and in proximity to energy development facilities), aquifer type (wells in the shallower aquifers have a higher priority that the deeper aquifers), and that have historic water quality data to better assess potential recent and future changes in groundwater quality.

Currently we have located 18 groundwater monitoring wells that are available to be sampled and are described in **Table 2** and **Figure 3**. We also have permission to sample the New Town municipal drinking water wells that are in the New Town Aquifer. Other groundwater wells to the south of New Town and in the Mandaree area need to be identified for future sampling events. All samples will be analyzed for the full suite of compounds listed in **Table 3**.

Task 1: Reports

Water-quality data will be collected each year from 2014 through 2019. In 2016, a USGS Scientific Investigations Report will be published that includes an interpretation of the data collected in the ambient monitoring program described in this QAPP and the data collected by the NDDH and the USACE from Lake Sakakawea. All data collected by USGS directly or from other sources for preparation of the Scientific Investigations Report will be provided to the tribe in a useable electronic format to support the tribe's water quality monitoring goals. The TAT Environmental Department will produce a data assessment report each year as part of their requirement for funding under the USEPA Clean Water Act Section 106 program.

Task 2: The various types of field data collected, meter calibrations, field measurements, streamflow measurements, groundwater levels, and groundwater well purging data will be recorded on standard USGS electronic Personal Computer Field Forms for the collection of surface water and groundwater-quality data (Wilde, F.D., 2008; Chapter A6, section 6.0.1.A). The field data collected and all constituents analyzed at the USEPA laboratory in Golden, Colorado will be entered by USGS into the USGS National Water Information Systems (NWIS) database accessible at http://waterdata.usgs.gov/nd/nwis/qw and by the TAT environmental department into the USEPA Ambient Water Quality Monitoring System (AQWMS) database in fulfillment of USEPA requirements for tribes receiving funding under section 106 of the Clean Water Act.

A7 Quality Objectives and Criteria

Several criteria need to be addressed in order to meet the quality objective of the project. These include:

Performance

Samples and related information will be collected, documented, preserved, packaged, maintained under custody and transferred to the laboratory as described in this QAPP to ensure that the data generated are complete, accurate, and of the type, quantity and quality required for their intended use. Laboratory procedures are controlled and performed by the EPA Region 8 Laboratory as described in the Laboratory QA Manual (**Appendix I**).

Measurement Criteria

All information to be collected must be in accordance with approved USGS methods as described in the USGS National Field Manual for the Collection of Water-Quality Data (http://water.usgs.gov/owq/FieldManual). This project is to establish current water quality conditions in streams and groundwater on the Fort Berthold Indian Reservation in 2014 and monitor long-term trends in water quality. The YSI Multi-meter 6920 V2-2 used for the in-situ field measurements shall have both pre and post sampling calibration performed each day. The post calibration results for conductivity and pH should be within 5 percent of their pre-sampling calibration values. Stream discharge data will be measured at the six streams described in section A6. The USGS stream discharge measurement criteria will be accepted for this project (Rantz, S.E., and others, 1982).

Acceptance Criteria

For EPA Region 8 analyses, target analytes, analytical methods, reporting limits, and acceptance criteria for selected analytes for this project are found in **Appendix 2.** Specific method descriptions and procedures are available from the EPA Region 8 Laboratory.

Any data not meeting the Laboratory acceptance criteria for a method will be flagged accordingly, by the Laboratory QA Manager. The Project accepts the laboratory acceptance criteria for Project use. The EPA Project Manager will review Laboratory flagged data, and quality control (QC) sample results. For field blanks, results should be non-detects. If there are detections of an analyte in a field blank, that analyte will be flagged for those analyte results for the sampling event. Replicate samples should compare favorable to the base sample where the replicate was taken. It is not expected that the replicate result be the same as the base sample, however; the replicate result should be within plus or minus 20 percent of the base value. If the replicate result exceeds that for an analyte, the EPA Project Manager will investigate (checking with the surface water sampling staff). Replicate samples should be taken alongside the base sample. Based on finding of the investigation, the base sample results may be flagged.

Stream discharge measurements and GPS measurements will be accepted by the USGS and TAT Project Manager if, the project SOPs have been followed in the collection/measurement process.

Project decisions will not be based on data results that are flagged. Flagged data will only be informational and are not to be used in decision making.

Data Quality Objectives

It is the policy of the USEPA and the TAT Environmental Division that the data quality objectives (DQOs) be developed for all environmental data collection activities. Data of known quality are essential to the success of any monitoring or sampling project. Data quality objectives are qualitative and quantitative statements that clarify the intended use of the data, define the type of data needed to support the decision, identify the conditions under which the data should be collected, and specify tolerable limits on the probability of making a decision error due to uncertainty in the data. DQO's are developed by data users to specify the data quality needed to support specific decisions. Sources of error or uncertainty include the following:

-Sampling error: The difference between sample values and *in situ* values from unknown biases due to collection methods and sampling design;

-Measurement error: The difference between sample values and *in situ* true values associated with the measurement process;

-Natural variation: Natural spatial heterogeneity and temporal variability in population abundance and distribution; and

-Error sources or biases associated with collecting, sampling, handling, storage, and preservation.

Methods and procedures described in this document are intended to reduce the magnitude of the sources of uncertainty and their frequency of occurrence, by applying the following approaches:

-use of standardized sample collection, handling, and analysis procedures; and

-use of trained scientists and technicians to perform the sample collection and handling activities.

The purpose of this project is to establish a long-term ambient water-quality monitoring program and propose acceptable standards for the groundwater and surface waters within the Fort Berthold Indian Reservation boundaries.

The objective of quality assurance for the measurement data is to insure confidence in the data used to establish water quality standards, to determine appropriate water use classifications, and to evaluate various environmental indicators.

Representative sampling, proper sample handling techniques, and appropriate data evaluation methods must be used.

A8 Special Training/Certifications

Staff leading and performing all field operations shall have appropriate water quality sampling training and field health and safety training. The USGS Project Manager is trained and provides training in the collection of water quality data. HAZWOPER training is not needed for the water quality sampling to be done in this project. The USGS field sampling crew has the necessary knowledge and experience to perform all field activities and provide the necessary training in data collection methods and field health and safety training to the TAT environmental staff. The Tribal Project Manager is responsible for documenting and maintaining the training in the project file and will confirm that the appropriate training has been taken by all staff involved in the project.

The EPA Region 8 Laboratory is NELAC Certified for Drinking Water and nonpotable water analyses. The latest EPA Region 8 Laboratory NELAC recertification date was on 10/1/2013. The certification is good until 9/30/2014.

A9 Documentation and Records

Sampling documentation is intended to provide the laboratory with sufficient information to successfully support field investigations. The laboratory will provide the EPA Project Manager with an electronic copy of the analytical report and analytical results. The project water quality and physical data will be permanently archived by the TAT Project Manager. The TAT Project Manager will perform data assessments and will maintain copies of reports produced from the assessments. The TAT Project Manager will also be responsible for uploading analysis results and measurements into the EPA AWQMS DATABASE Warehouse as permanent data records.

The QAPP is prepared and maintained by the TAT Project Manager in collaboration with the USGS Project Manager. It is responsibility of the TAT Project Manager to review and update the QAPP as required and to ensure that individuals on the Distribution List are provided a copy of the latest version of the QAPP and any revisions on timely basis. Reviews and revisions of the QAPP will be performed in accordance with the most recent EPA Region 8 Quality Management Plan (QMP). The approved Quality Assurance Project Plan will be stored in project files which are maintained by the EPA and TAT Project Managers.

Documents and Records for this project include information generated in the field (e.g., field log books and chain-of-custody forms), assessment reports, progress/status reports, and a final project report. Project reports will be provided to the EPA Project Manager and to the TAT Project Quality Assurance Officer.

The final project report will describe the project, personnel involved, the sampling events and accomplishments (samples collected, field measurements taken, the development of the Project benchmarks for selected analytes, and the results of the project. The description of the development of the Project benchmarks shall include the sources used and the rationale for the benchmark determination. The final project report will be delivered to the EPA Project Manager and the TAT Project Quality Assurance Officer.

Dedicated Field Book

A field notebook will be maintained by the USGS Project Manager and the TAT Project Manager. During each sampling event, field parameter values are logged on project field sheets which are then inserted in the book. The field book is also used to log any activity which occurred during the sampling event. Field log book and other information will be kept electronically and printed for back up. Calibration information for each day's sampling event is kept in a calibration book that is kept with the specific multi-probe meter/sonde unit.

The following information will be recorded for each measurement on U.S. Geological Survey Surface-Water Quality Field Notes **(Appendix III)** and U.S. Geological Survey Groundwater Quality Field Notes **(Appendix IV)**:

- Name of stream site location to correctly identify the sample location. The location information should include the site ID, site name (including the stream) and the latitude / longitude coordinates.
- Date and time, person(s), types of meters and unit #
- General weather and stream conditions
- Water temperature and other field parameters
- Other pertinent information, including unexpected events or deviations from SOPs

Dedicated Project Files

Dedicated project files contain detailed information concerning sample sites for each project. Each folder contains at minimum of station photos, a map of the location, and dedicated project field sheets. Field sheets are developed for each sampling event

Chain of Custody Forms

Chain-of-custody forms are utilized to trace the transfer of samples from sampling staff to the analytical laboratory staff. An example of the EPA Region 8 Laboratory chain of custody form is in **Appendix V**.

B1 Sampling Process and Design

For baseline sampling, the six major creeks that drain into Lake Sakakawea being sampled are listed in **Table 1** and shown in **Figure 2 and 3**. Water-quality samples from the Bear Den Creek, Squaw Creek, Moccasin Creek, Shell Creek, East Fork Shell Creek, and Deepwater Creek locations will be analyzed for the full suite of compounds listed in **Table 3**. Water-quality samples will be collected from springs during each sampling event and will be analyzed for the full suite of compounds listed in **Table 3**. The flow of each spring (discharge) will also be measured each time a water-quality sample is collected. The springs to be sampled will be selected during the first sampling event in April 2014. A field reconnaissance is needed to accurately assess the proximity of the springs to land use activities. Water-quality samples will be collected from groundwater wells by USGS personnel and the TAT Environmental Department and will be analyzed for the full suite of compounds listed in **Table 3** twice each year, once in April and once in August at the same time the streams are sampled. Groundwater levels also will be measured each time a water-quality sample is collected. It is not possible to measure groundwater levels in domestic wells because they are sampled at the tap and there is no access to the well to be able to measure groundwater levels.

Sensitivity

The analytical methods selected for this project have the lowest level reporting limits available from the EPA Region 8 Laboratory for the analytes selected. Since there are no project resources available to consider other laboratories for analysis, other methods were not considered. Nonetheless, the EPA Region 8 Laboratory is performing analysis for many other oil and gas related projects, including projects outside of Region 8. Since the other projects have deemed the methods sensitive enough to meet the project objectives, this project considers the methods sufficient. The Project Manager spent many hours discussing the analytical methods with the EPA Region 8 chemists who will be performing the analyses. Based on those discussions, the Project Manager is confident that the methods are appropriate and sensitive enough to meet the project objectives.

Accuracy

Accuracy is a measure of the overall agreement of a measurement to a known value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations. Accuracy is expressed in terms of precision and bias.

The Project Manager will optimize the accuracy of field measurements by using repeatable field practices following standard operating procedures, ensuring that field instruments/equipment are calibrated and maintained according to the manufacturer's instructions, and by following the manufacturer's instructions for operation of the equipment.

GPS location measurements of latitude and longitude coordinates should be within 15 meters of the actual sampling site location coordinates. Coordinates in the QAPP are derived from image interpretation in GIS. GPS coordinates in the field should accurately identify a sample within 15 meters of its true location. This geospatial information will be collected and managed in accordance with EPA and federal requirements and guidance, including EPA Policy 2131 and EPA CIO 2131-P-01-0.

Precision

Precision is a measure of agreement among repeated measurements of the same property under identical, or substantially similar, conditions. Precision is normally reported in terms of standard deviation, but since the number of measurements is small, for this project it will be reported as relative percent difference.

Relative percent difference (RPD) is calculated by:

$$\text{RPD (\%)} = 100 \times \{[X1 - X2]/X_{ave}\}$$

Where: X_1 = concentration observed with the first detector or equipment;
X_2 = concentration observed with the second detector, equipment, or absolute value; and
X_{ave} = average concentration = ((X1 + X2) / 2)

Information regarding precision will be collected in the field by collecting a field duplicate sample and duplicating field measurements at one site per sampling event. Laboratory precision is addressed in the methods and Laboratory QA Manual. The information will be reviewed and evaluated as part of the data review, verification, and validation process.

Bias

Bias is the systematic or persistent distortion of a measurement process that causes error in one direction (i.e., the expected sample measurement is different from the sample's true value). Bias will be estimated using matrix spike analysis as described in the laboratory procedures and Laboratory QA Manual, reported in terms of percent recovery.

Percent Recovery (%R) is calculated using the following equation:

$$\%R = 100 \times (X - B) / T$$

Where: X = measured concentration of the spiked sample;
T = theoretical true concentration of the spiked sample; and
B = background concentration of the unspiked sample.

Representativeness

Representativeness is the measure of the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. The project information is intended to represent the oil and gas discharge contaminant distribution and magnitude trends downstream of the discharges at varying distances. Samples will be collected in two different events in order to have at least two samples per site. The two events will capture some variations (seasonal and maybe flow) using grab sampling techniques for all streams/rivers that are monitored. This sampling protocol was selected because grab sample methods are a practical means of collecting samples for small streams with homogenous water quality. Samples will be collected from downstream locations to upstream locations to minimize the impact of sampling operations on the data. Variable factors that will be taken into consideration during this project will be stream peak flows and precipitation reports for the targeted sites.

As was discussed in the second paragraph of this **B1** section, the G-52, Muddy Creek sampling site is considered to be representative of the other sub-watershed sampling sites. The surface water sampling lead is asked to evaluate its representative nature when sampling occurs.

Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system. It is calculated as a ratio of the amount of valid or usable data collected to the amounts of valid or usable data planned. The goal for the project is 100% sample collection for all fixed sites; lesser completeness results will not fatally affect the project outcome.

Percent Completeness (%C) is calculated using the following equation:

$$\%C = V \times 100 / T$$

Where: %C = percent completeness,
V = number of planned measurements determined valid, and
T = total number of measurements.

Comparability

Comparability is the measure of the confidence with which one data set or method can be compared to another. The comparability of project information is enhanced by the use of standard analytical methods, field practices and procedures. Documentation of the planning, implementation and assessment phases of the project also provides additional information to evaluate the comparability of data.

B2 Sampling Methods

All water quality data will be collected in accordance with approved USGS methods as described in the USGS National Field Manual for the Collection of Water-Quality Data (http://water.usgs.gov/owq/FieldManual) in chapters A1-A6 and A9. This field manual describes procedures for equipment cleaning and decontamination between sample and the proper disposal of decontamination products. The citations in the following discussion are for specific chapters in the USGS Field Manual. A list of additional support equipment needed for field sampling is found in Appendix VIII. No continuous monitoring or in-situ data will be collected.

B2.1 Stream and Drainage Sampling

Water-quality samples will be collected using the equal-width-increment (EWI) method. For the EWI method, the stream cross section is divided into a number of equal-width increments. Samples are collected by lowering and raising a sampler through the water column at the center of each increment (this sampling location is referred to as the vertical). The combination of the same constant transit rate used to sample at each vertical and the isokinetic property of the sampler results in a discharge-weighted sample that is proportional to total streamflow. If stream velocities are less than 1.5 cubic feet per second, a grab sample will be collected from the center of the stream channel. Samples collected with either method will be composited into a churn splitter and samples are processed from the churn splitter. Sample processing refers to the measures taken to prepare and preserve a water sample as or after it is collected and shipped for laboratory analysis. A description of the equipment and procedures to be used in collecting water-quality samples from streams are found in the USGS National Field Manual (NFM) (USGS, 2006, Chapter A4, section 4.1). The sample processing procedures are described in section B3.

B2.2 Groundwater Sampling

The procedures for sampling groundwater wells also are described in the USGS NFM (USGS, 2006; Chapter A4, section 4.2). Domestic groundwater wells are described in the USGS NFM (USGS, 2006; Chapter A4, section 4.2.4.A), and the NDSWC groundwater wells procedures are described in the USGS NFM (USGS, 2006; Chapter A4, section 4.2.4.B). Groundwater levels will be measured with a Solinst Model 101 Water Level Meter according to the procedures described in (USGS, 2006; Chapter A4, Appendix A4-B). The locations are shown in **Table 2** and **Figure 3**.

B2.3 Spring Sampling

Water-quality samples will be collected from the five springs as a grab sample at the point where the spring discharges from the ground. The procedure for collecting a grab sample and the method for measuring spring discharge are found in (USGS, 2006; Chapter A4, section 4.1.3B; Rantz, S.E., and others, 1982). These locations will be selected during this year's reconnaissance. Criteria that will be used for selection will closely resemble that used for drainage site selection.

B2.4 Field Properties and Streamflow

The procedure used for field measurements of pH, specific conductance, water temperature, and dissolved oxygen are described in (Wilde, F.D., variously dated; Chapter A6, sections 6.0-6.8*)*. Alkalinity titrations, from which bicarbonate (HCO_3^-) and carbonate (CO_3^{2-}) concentrations are calculated, will be completed in the field using filtered-water samples within 2 hours of sample collection (Rounds, S.A., 2012, Chapter 6.6). A HachTM digital-titration kit using 0.16 N or 1.6 N sulfuric acid titration cartridges, calibrated Radiometer pH meter, and magnetic stirrer will be used for the alkalinity titrations. Streamflow measurements and spring discharge will be measured according to the procedure described in (Rantz, S.E, and others, 1982*)*.

B3 Sample Handling and Custody

All water quality samples (stream, groundwater, and spring) will be processed in the field using standard USGS techniques (Wilde, F.D. and others, variously dated; Chapter A5, section 5.0-5.2). Samples will be processed in a dust-free processing chamber using "clean hands" procedures. Samples to be analyzed for dissolved constituents will be filtered through a 0.45-micrometer (µm) pore-size disposable capsule filter with a Geotech easy-load geopump. Trace-element samples will be preserved with 7.7 normal (N), ultrapure nitric acid. Table 5 summarizes the bottle type, sample preservation requirements, and holding times for each category of analytes measured in water samples.

Upon completion of sample processing all samples will be placed in an iced cooler at a temperature of 4 °C. Coolers will be transported from the Fort Berthold Indian Reservation to the USGS North Dakota Water Science Center in Bismarck, North Dakota by USGS personnel at the completion of each sampling event. All coolers will be shipped from the USGS North Dakota Water Science Center to the EPA Region 8 Laboratory in Golden, Colorado by Federal Express. An EPA chain of custody form (Appendix V) will be completed and submitted with the samples.

B4 Analytical Methods

All water quality samples that are collected from streams, groundwater wells, and springs will be analyzed for the full suite of compounds listed in **Table 3** at the EPA Region 8 Laboratory in Golden, Colorado. Analytical methods, containers, sample preservation, and holding time requirements are described in **Table 4**. Analytical methods and specific analytes are identified in Appendix 1. EPA Region 8 Laboratory procedures are controlled and performed by the EPA Region 8 Laboratory in accordance with the Region 8 Laboratory QA Manual (**Appendix 1**). From the time of sample receipt by the laboratory, report turnaround time is 60-90 days.

Laboratory Information:	**Laboratory Project Contact:**
EPA Region 8 Laboratory	Jack Sheets
16194 W. 45th Drive	EPA Lab Project Manager
Golden, CO 80403-1790	USEPA Region 8 Laboratory
(Phone) 303/312-7770	sheets.jack@epa.gov
(FAX) 303/312-7800	(Phone) 303/312-7793

B5 Quality Control Requirements

To satisfy the data quality objectives, the quality assurance/quality control (QA/QC) plan for this ambient monitoring program includes the use of approved USGS methods for the calibration of field property meters and the collection and analysis of surface and ground water samples, as described in subsequent sections. Additional QA/QC methods will include the computation of a cation/anion balance for each sample, a comparison of the dissolved to total metal ratios, the collection of field blanks and field duplicates, and the addition of matrix spikes to the trace element samples as described in Mueller and Titus (2005) and will be described briefly.

In any water-quality study two types of samples are needed: environmental samples and quality-control samples. Environmental samples fulfill the scientific objective(s) of the study. Quality-control samples provide estimates of the bias and variability of the environmental data. Field blanks are samples that are intended to be free of the analyte(s) of interest and are analyzed to test for bias from the introduction of contamination into environmental samples in any stage of the sample-collection and analysis processes. Field replicates are a group of samples that are collected in a manner such that the samples are thought to be essentially identical in composition and are used to estimate the variability of the sample-collection and analysis process. Field blanks and field replicates are collected in the same manner as the environmental samples. Matrix spikes are environmental samples that will be fortified (spiked) in the laboratory with a known mass of the analyte(s) of interest. These samples are used to assess the potential bias for organic and inorganic analytes in a particular sample matrix. In spiked samples, the bias is estimated by calculating the percentage of each added analyte that is measured (recovered) in the sample. Bias can be either positive or negative with possible recoveries greater than or less than 100 percent. Because these samples are spiked at the laboratory, a separate sample aliquot will be collected and identified for this purpose. Custom spike solutions have been determined for selected organic and inorganic compounds identified by USEPA (U.S. Environmental Protection Agency, 2011 a).

Field Blank Water

Water for the field blanks will be provided by the USGS North Dakota Water Science Center. The inorganic and organic water is purchased from approved USGS National Field Supply

Services (NFSS) and includes a certificate of analysis. The NFSS Quality Assurance Surveillance Plan can be accessed at http://wwwnwql.cr.usgs.gov/qas/QASP.pdf.

Cation/Anion Balances

The accuracy of the analysis of major dissolved ions will be evaluated by calculating a cation/anion balance for each sample. A fundamental principle of solution chemistry is that a condition of electroneutrality exists for the major ions dissolved in water, which means that when measured in milliequivalents per liter, the sum of the positive charges equals the sum of the negative charges. The equation used to calculate a cation/anion balance is:

$$\text{cation/anion balance} = \frac{(\text{sum of the cations} - \text{sum of the anions})}{(\text{sum of the cations} + \text{sum of the anions})} \text{ X } 100 \quad (1)$$

Ideally the result of this calculation should equal zero but in practice some deviation from zero is acceptable. If significant deviation from zero occurs, there must be either analytical errors in the analytical measurement or the presence of an ionic specie or species at significant concentrations that were not included in the analysis. The criteria used for the determination of an acceptable cation/anion balance in this study will be ± 5%. Any sample that falls outside of this range will be submitted for reanalysis. The cations to be used in the above equation are dissolved calcium, magnesium, sodium, and potassium. The anions to be used in the above equation are dissolved bicarbonate, carbonate, sulfate, chloride and nitrate. The USGS Water-Quality Data Management Lead will develop an Excel spreadsheet that the TAT Project Manager will use to calculate the cation/anion balance for each sample. An analysis of the cation/anion balance cannot be used as the only means of detecting measurement error because an acceptable cation/anion balance may occur in situations where large errors in the individual ion analyses balance one another. The cation/anion balance also does not evaluate the quality of the analysis for dissolved and total trace elements. Therefore, the results of the analysis of dissolved/total trace element ratios, field blanks, field replicates, and matrix spikes will be discussed subsequently. During the next revision of the QAPP, the spreadsheet that is developed will be included in the QAPP. The information will include the equations being used in the spreadsheet to generate the analysis.

Trace Element Dissolved/Total Ratios

An evaluation of the quality of the trace element analysis involves a comparison of the ratio of the concentration of the dissolved fraction to the concentration of the total fraction. Ideally, the concentration of the total fraction of a trace element should always be greater than or no less than equal to the dissolved fraction of the trace element. However, due to variability that may occur as a result of sample collection, processing, transport, and analysis, the dissolved fraction may sometimes be greater than the total fraction. This situation commonly occurs at low concentrations. Therefore, the following criteria will be used to evaluate the quality of the analysis of trace elements. When sample concentrations are less than 1 mg/L a rerun of both the dissolved and total fractions will be requested when the concentration of the dissolved fraction exceeds the concentration of the total fraction by more than the lab reporting limit (LRL) of the less precise of the two methods. For example, if the LRL for dissolved Cu is 0.23 µg/L and the LRL for total Cu is 0.6 µg/L, dissolved Cu may exceed total Cu by 0.6 µg/L. If sample concentrations are equal to or greater than 1.0 mg/L, than the concentration of the dissolved fraction can exceed the concentration of the total fraction by less than 10%. After each sampling event the ratio of the concentration of the dissolved fraction to the total fraction will be checked according to this criteria and a reanalysis of the dissolved and total fraction for any sample which failed this comparison will be requested of the NWQL. All dissolved and total trace element concentration data will not be entered into the USGS NWIS database until these criteria are satisfied.

Field Blanks and Field Duplicates

Field blanks and field duplicates will be collected during this ambient monitoring to quantify the errors involved in collecting, processing, transporting, and analyzing the samples. Any measurement has an error associated with it that cannot be eliminated, but the error can be quantified so that appropriate interpretations of the environmental data can be made. Bias and variability are two components of the error associated with any water-quality measurement. Bias is the systematic error inherent in a method or measurement system and may be either positive (contamination) or negative (loss). Variability is the random error in independent measurements that results from repeated application of the measurement process under specified conditions.

Once a data set is established with an estimated amount of bias and variability, it is necessary to determine how the bias and variability affect the interpretation of the environmental data. Thus, the analysis of quality-control sample data supports the interpretations of the environmental data by establishing, with a known level of confidence, the amount (if any) of sample contamination that has occurred during the study and by establishing the range of variability in the quality-control sample data relative to the range of variability in the environmental data.

Analysis of Field Blanks

Under ideal conditions any contamination present in field blanks would be so small that concentrations would be less than the detection limit. In practice, although concentrations measured in many field blanks are less than the detection limit, some blanks contain concentrations greater than the detection limit. Therefore, as stated in Mueller and Titus (2005), "The objective in analyzing data from blanks is to determine the amount of contamination that is not likely to be exceeded in a large percentage of the water samples represented by the blanks. This objective can be achieved by constructing an upper confidence limit (UCL) for a high percentile of contamination in the population of water samples that includes environmental samples and blanks. This UCL is the maximum contamination expected in the specified percentage of water samples. For example, the 95-percent UCL for the 90th percentile of concentrations in blanks is the maximum contamination expected in 90 percent of all water samples. The 95-percent confidence level indicates there is only a 5-percent chance that this contamination has been underestimated. Another way to express this is that we are 95-percent confident that this amount of contamination would be exceeded in no more than 10 percent of all samples (including environmental samples) that were collected, processed, and analyzed in the same manner as the blanks."

In calculating the UCL for the blank data all estimated values and values that were detected but were within the range of two or more detection limits (if there are multiple detection limits) will be censored to the highest detection limit. An example of how to interpret this data is the following excerpt from Naftz and others (2011). "A review of the field blank data in tables 4 and 5 shows that all the blanks analyzed for dissolved beryllium, boron, cadmium, chloride, cobalt, fluoride, iron, lithium, nitrate + nitrite, selenium, silver, sodium, sulfate, thallium, and uranium and total selenium were reported as less than the detection limit. Thus, contamination by each of these analytes is estimated with about 92-percent confidence to be no greater than the detection limit in at least 70 percent of all samples. The 92-percent confidence level indicates that there is only an 8-percent chance that this contamination has been underestimated. For those analytes that had measurable concentrations in the blanks, we are 92-percent confident that the amount of contamination listed in table 4 would be exceeded in no more than 30 percent of all samples. For example, for dissolved uranium there is 92 percent confidence that contamination is no greater than the detection limit of 0.02 µg/L in at least 70 percent of all samples. For total uranium there is 92 percent confidence that contamination is no greater than 0.024 µg/L in at least 70 percent of all samples. Another way to express this is that contamination by total uranium is estimated, with 92-percent confidence, to exceed 0.024 µg/L in no more than 30 percent of all samples".

This amount of contamination can then be compared to environmentally important concentrations of each analyte to determine the likelihood that contamination has affected interpretation of the environmental data. Mueller and Titus (2005) state that "in general, if potential contamination is less than 10 percent of a measured value, the effect of contamination bias on that measured value can be ignored." An example of this interpretation is provided by another excerpt from Naftz and others (2011). "The detection limit for all of the analytes that were never measured above the detection is at least 10 times less than the environmental concentrations measured in this study or EPA drinking water MCL's. For example, the detection limit of dissolved uranium (0.02 µg/L) is 1,500 times less than the EPA drinking water MCL of 30 µg/L. So even if contamination were ≥ 0.02 µg/L in 30 percent of all samples the contamination would have to be two orders of magnitude greater than this value for potential bias to affect the interpretation of the uranium environmental data".

Analysis of Field Replicates

The field replicate data will be analyzed to assess the amount of variability present in the environmental data by calculating a 95 percent confidence interval about a single sample and determining the minimum significant difference that can be detected between any two individual measurements using the equations given in Mueller and Titus (2005). These calculations involve calculating a standard deviation for each field replicate pair and examining graphs of the standard deviation of each replicate pair as a function of the average concentration of each field replicate pair to determine if the standard deviation is constant over the range of concentrations measured. If the relation between standard deviation of each replicate pair is constant over the range in concentration measured for each constituent or only a weak relation with concentration exists the average standard deviation of the replicate pairs for each constituent will be substituted into the following equation to calculate a 95 percent confidence interval about a single sample:

$$C_{interval} = C_{sample} \pm Z_{0.95}SD \qquad (2)$$

where:

$C_{interval}$ is the confidence interval about a single measurement = 100(1-α);
C_{sample} is the concentration of a single sample;
SD is the average standard deviation of the replicate pairs; and
$Z_{0.95}$ is the statistic for the 95-percentage point of the standard normal curve = 1.96

If the value of the replicate pair standard deviation increases as concentration increase then for concentrations in this high range the standard deviation (SD) in equation 2 becomes:

$$SD = C_{sample}(\text{relative standard deviation of the replicate pairs; RSD/100})$$

If one of the replicate pairs is reported as less than the reporting limit but the other had measurable amounts of a constituent reported, the sample with a value of less than the reporting limit will be assigned a value of one-half the reporting limit to perform the calculation. The 95 percent confidence interval data about a single sample can be interpreted in the following manner. There is 95 percent confidence that the true value of any individual measurement for any constituent will lie within the range calculated with equation 2.

To determine the minimum significant difference that can be detected between any two individual measurements the following formula was used:

$$\Delta C \text{ (difference in concentration between two samples)} \geq 1.96 * \sqrt{2} * SD \qquad (3)$$

If the difference in concentration between any two samples is equal to or greater than this amount there is a 95 percent probability that the difference is significant.

Matrix Spikes

An aliquot from one filtered and one unfiltered sample will be collected during each sampling event will be spiked with trace metals at the US EPA Region 8 Laboratory in order to evaluate whether or not the sample matrix (the overall chemical composition of the sample) affects the quality of the metal analyses. Percent recoveries computed on spiked and unspiked samples for all trace elements will be compared to the USEPA percent recovery allowable limits for laboratory spiked environmental samples analyzed via ICP-MS for total metals (Region 8 Lab 200.7/6010) and dissolved metals (Region 8 Lab 220.8/6020).

B6 Field Instrument/Equipment Testing, Inspection, and Maintenance

Field Instrumentation Calibration

Field equipment will be operated, tested, inspected, maintained and calibrated according to manufacturer instruction and requirements. See **Table 3** for a list of field sampling equipment. Maintenance logs will be kept; each piece of equipment will have its own maintenance log. The log will document any maintenance and service of the equipment. The USGS Project Manager is responsible for assuring that testing, inspection, and maintenance of equipment has been performed and ensuring that repairs are effective in correcting deficiencies. Spare parts will be ordered from equipment supply vendors as needed by the USGS Project Manager, if applicable. Field properties (pH, specific conductance, water temperature, and dissolved oxygen concentration) will be measured at the sampling site with a multi-meter instrument (Yellow Springs Instrumentation 6920 V2-2) that is calibrated each day before use according to (Wilde, F.D., ed., variously dated, Field measurements: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A6) which includes section 6.0 – 6.8 and covers the instrument calibration of all field measurements for this project.

B7 Field Instrument/Equipment Calibration and Frequency

Field instruments/equipment will be calibrated as described by the manufacturer. Calibration records will be included in the project field log book. If any deficiencies are found the field team will contact the products supplier and get assistance on correcting the deficiency prior to use in the field. The USGS Project Manager is responsible for ensuring that equipment is calibrated and documented as required.

B8 Inspection/Acceptance Requirements for Supplies and Consumables

The US EPA Region 8 QA Laboratory Manual describes the type of sample containers and preservatives needed and assures they are acceptable for use. All supplies are inspected by a member of the field sampling team to ensure that materials are delivered intact. If materials are not delivered intact, they are rejected for use in sampling programs. Sample containers are provided by the laboratory. All supplies are to be stored in protective containers and out of direct sunlight. All supplies are marked with an identification sticker that coordinates to the program that purchased the supplies. All purchases must be approved by the USGS Project Manager and are reviewed on a case by case basis.

B9 Data Acquisition Requirements (Non-Direct Measurements)

Non-direct information may include climatic and weather data from the National Weather Service, stream discharge data from U.S. Geological Service (USGS) gauging stations, water quality data from the USGS NAWQA program. GIS-derived latitude and longitude coordinates for sampling sites. This information is deemed to be reliable and adequate for project use, as provided.

Existing data used for the retrospective report were collected by the NDDH according to their Quality Assurance Project Plans for the Lake Sakakawea Water Quality Monitoring Project for years 2003-2013 and by USACE according to their Quality Control Plan for Ambient Monitoring of the Missouri River Mainstream Reservoirs for years 2003-2013.

Weather and climate data is used to evaluate streams conditions at the time of sampling and to assess potential runoff conditions within a week prior to sampling. Weather data will be accessed from the NOAA National Climatic Data Center at:

http://www.ncdc.noaa.gov/cdo-web/

Coordinates, derived from GIS images, for sampling sites will be verified in the field with GPS. Any information regarding discrepancies or errors in this information will be provided to the USGS Project Manager.

USGS water data has a thorough quality system associated with it. The key website for quality information for USGS water quality data is:

http://water.usgs.gov/owq/quality.html

The descriptions of the USGS water quality monitoring methods is found at:

http://water.usgs.gov/owq/methods.html

All metadata for GIS-derived latitude and longitude coordinates will be collected in conformance with Federal and EPA Geospatial policy as described in:

http://www.epa.gov/geospatial/docs/2131.pdf

To determine targeted sampling locations, dates, quantities, and hazards/chemical characteristics of the spills the following weblink will be used:

http://www.gagecartographics.com/spills.

B10 Data Management

Field information is reviewed and checked by the field sampler before transfer to the USGS Project Manager. Original data sheets, copies of logbooks page entries for the project, and other field information will be provided to the USGS Project Manager. Backup copies of data will be maintained by the USGS Project Manager in project files by for five years, or longer if required by grant requirements or statute. Analytical results provided by the US EPA Region 8 Laboratory in an electronic format will be managed under the direction of the TAT Project Manager and the USGS Project Manager. The data will be transmitted to EPA's AWQMS database by the TAT Project Manager via WQX transfer tools. Backups are to be made on external hard drives. The TAT Project Manager will provide the data electronically for backup to the EPA Project Manager after each sampling event.

OVERALL FLOW FOR DATA MANAGEMENT PROCEDURES

Sample Process Step	Tracking Tool	Primary Responsibility
Preparation for sampling	Supply inspections, field collection check list, equipment calibration notebook check	USGS Project Manager
Field data collection	Field book, station file, sample label, sample tracking form	USGS Project Manager
Sample transfer	Chain of custody form	TAT Project Manager and USGS Project Manager
Analytical determinations	Laboratory results, laboratory QA/QC	Laboratory staff, EPA Project Manager
Data review and validation	Data review reporting for each sampling event	TAT Project Manager and USGS Project Manager
Corrective action (if required)	Corrective action reporting to QA Manager	TAT Project Manager and USGS Project Manager
Data entry and management	Data management form	USGS Project Manager and TAT Project Manager

C1 Assessments/Oversight and Response Actions

Assessment activities and corrective actions have been identified to ensure that sample collection activities are conducted as prescribed and that the measurement quality objectives and data quality objectives established by this QAPP are met. The QA program under which this project will operate includes performance and system audits with independent checks for the data obtained from sampling activities. Either type of audit could indicate the need for corrective action. The essential steps in the program are as follows:

Immediate corrective actions from the part of normal operating procedures and are noted on the project field and laboratory recording forms and will be the responsibility of the field personnel. Problems not solved this way may require more formalized, long term corrective action. In the event that quality problems requiring attention are identified, the TAT Project Manager and/or the TAT Project Quality Assurance Officer will determine whether attainment of acceptable data quality requires either short or long term actions.

Communication and oversight will proceed from TAT Project Manager to the TAT Project Quality Assurance Officer and will also include a review by a level II QA staff member outside the Water Quality Division. The TAT Project Manager will be available throughout the entire sampling period to address questions and receive communications of sampling status from the field personnel. Field personnel will communicate the status of sampling activities to the TAT Project Manager and/or TAT Project Quality Assurance Officer on a weekly basis. During this time, field personnel will communicate any sampling difficulties encountered during sampling and corrective actions will be taken. In most cases, field personnel will initiate corrective actions when a problem is immediately identified and note the problem and corrective action in his/her logbook. In the event the problem cannot be corrected immediately, field personnel will contact the TAT Project Manager and/or the TAT Project Quality Assurance Officer to determine the best way to rectify the problem to obtain accurate and usable data. When corrective actions have been taken and a sufficient time period has elapsed that allows a response, the response will be compared with project goals by the TAT Project Quality Assurance Officer. The TAT Project Quality Assurance Officer will verify that the corrective action has been appropriately addressed to eliminate the problem. The TAT Project Quality Assurance Officer has the authority to stop work on the project if problems affecting data quality are identified that will require extensive effort to resolve. When the TAT Project Manager and/or TAT Project Quality Assurance Officer are contacted with a problem, field personnel, the TAT Project Manager and/or the TAT Project Quality Assurance Officer should keep a record of the problem and the corrective action taken.

Performance audits are qualitative checks on different segments of project activities, and are most appropriate for field sampling and laboratory analysis activities. A field audit of field sampling activities will be conducted at least once during the project by the TAT Project Quality Assurance Officer. This audit will be conducted early during the project field season in case any problems are identified, so they can be corrected quickly to minimize the possibility of compromising data. Field audit techniques include checks on sampling equipment and the review of sampling methods.

System audits are qualitative reviews on project activity to check that overall project quality is functioning and that the appropriate QC measures identified in the QAPP are being implemented. The TAT Project Quality Assurance Officer will conduct an annual internal system audit during the project and report all deficiencies during annual reporting. The multi-year QAPP will undergo an annual review and update for changes made during the year and for the new work plan using the Region 8 QA Document Crosswalk link:

http://www2.epa.gov/sites/production/files/region8/qa/EPAR8_QA_DocumentReviewCrosswalk.docx.

C2 Reports to Management

Problems and corrective actions identified by the field personnel will be reported to the TAT Project Manager and/or TAT Project Quality Assurance Officer each week during the field season. Both small and significant problems identified by the field personnel as well as problems and corrective actions identified by the TAT Project Manager and/or TAT Project Quality Assurance Officer during the field audit will be reported as part of annual reports. These problems of all levels will be put into the field folder for future reference if the problem arises again at a later date. The annual review and revision of the QAPP and completed crosswalk will be submitted to the Tribal Project Manager for EPA approval prior to the start of data collection for the upcoming year.

D1 Data Review, Validation, and Verification

Data review and validation services provide a method for determining the usability and limitations of data, and provide a standardized data quality assessment. All field and laboratory report forms will be reviewed by the TAT Project Manager, while all sample custody forms for chemical analysis will be reviewed by the TAT Project Quality Assurance Officer for completeness and correctness. The TAT Project Manager will be responsible for reviewing all data entries and transmittals for completeness and adherence to QA requirements. Data quality will be assessed by comparing entered data to original data and by comparing results with the measurement performance criteria summarized in Section D.2 of heading I to determine whether to accept, reject, or qualify the data. Results of the review and validation processes will be reported to the TAT Project Quality Assurance Officer.

D2 Verification and Validation Methods

The tribe in cooperation with the USGS will provide a project-level verification and validation of all data based on the project data quality objectives to determine the usability of data, including qualifying data, as needed. The TAT Project Manager will review all field and laboratory record forms. The TAT Project Quality Assurance Officer will review a minimum of five percent of field and laboratory record forms and all of the sample custody forms for chemical analysis. Any discrepancies in the records will be reconciled with the field personnel and recorded in the logbook.

The submission of samples to the US EPA Region 8 Laboratory will include an analytical services request form documenting the site location, sampling date and time. The US EPA Region 8 Laboratory will report results according to the Region 8 QA Plan section 4.10 to the TAT and USGS. The TAT Project Quality Assurance Officer, in consultation with the USGS will determine whether or not to proceed with the analysis of that sample or constituent.

D3 Reconciliation with Data Quality Objectives

After each sampling event, as soon as the laboratory analyses are made available to USGS and TAT personnel, a calculation of a cation/balance and the total/dissolved trace element ratio for each sample will be done. Any sample not meeting the criteria for either of these calculations will be reanalyzed or qualified using qualifier codes supplied in AWQMS and WQX. A list and description of data qualifiers for AWQMS (Ambient Water Quality Monitoring System) and WQX (Water Quality Exchange) are listed in the database which is password protected. A screen shot of the qualifiers can be found in Appendix IX

The field blank and field duplicate data will be analyzed in two ways. First, the calculations for the field blanks and field duplicates will be done at the end of each year to check on the level of bias and the variability. At the end of the project all the field blank and field duplicate data will be compiled and analyzed to give an overall measure of bias and variability of all data collected during the project. However, the field blank and field replicate data will be reviewed after each sampling event and if individual field blanks and field duplicates exceed acceptable levels (contamination or bias exceeding 10% of environmental concentrations and variability greater than 20%) an attempt will be made to determine the cause of the contamination and/or high variability. At a minimum data verification and validation will include analyzing source solution blanks, equipment blanks, trip blanks, and reviewing EPA QC data.

E Literature Cited

Dingman, R. J., and Gordon, E.D., 1954, Geology and ground water resources of the Fort Berthold Indian Reservation North Dakota: U.S. Geological Survey Water-Supply Paper 1259, 115 p.

Mueller, D. K., and Titus, C. J., 2005, Quality of nutrient data from streams and ground water sampled during water years 1992–2001: U.S. Geological Survey Scientific Investigations Report 2005-5106, 27 p.

Naftz, D.L., Ranalli, A.J., Rowland, R.C., and Marston, T.M., 2011, Assessment of potential migration of radionuclides and trace elements from the Whiet Mesa uranium mill ot the Ute Mountain Ute Reservation and surrounding areas, Southeastern Utah: U.S. Geological Survey Science Investigations Report 2011-5231, 146 p.

NDDH, June 2009. Quality Assurance Project Plan for the 2009 Lake Sakakewa Water Quality Monitoring Project, Environmental Health Section, North Dakota Department of Health, Bismarck, North Dakota.

NDDH, June 2012. Quality Assurance Project Plan for the 20012 Lake Sakakewa Water Quality Monitoring Project, Environmental Health Section, North Dakota Department of Health, Bismarck, North Dakota.

Rantz, S.E, and others, 1982, Measurement and computation of streamflow: Volume 1. Measurement of stage and discharge and Volume 2. Computation of discharge: U.S. Geological Survey Water-Supply Paper 2175, 631 p.

Stevenson, D.L., ed., 2012, Quality management system, U.S. Geological Survey National Water Quality Laboratory: unpublished U.S. Geological Survey Report, version 2.0, 3 January 2012, chapters variously paged, accessed December 10, 2012, at http://wwwnwql.cr.usgs.gov/USGS/elec/QMS_v2.pdf

USACE, March 2012. Sampling and Analysis Plan for 2012 Ambient Monitoring of the Missouri River Mainstem Reservoirs, Project Number: AMB-MAINST-009, U.S. Army Corps of Engineers - Omaha District

U.S. Environmental Protection Agency, 1994a, Method 200.8: Determination of trace elements in waters and wastes by inductively coupled plasma-mass spectrometry, U.S. Environmental Protection Agency data available online at URL http://www.epa.gov/waterscience/methods/method/files/200_8.pdf

U.S. Environmental Protection Agency, 1994b, Method 200.7: Determination of metals and trace elements in water and wastes by inductively coupled plasma-atomic emission spectrometry

U.S. Environmental Protection Agency data available online at URL http://www.epa.gov/waterscience/methods/method/files/200_7.pdf

U.S. Geological Survey, 2006, Collection of water samples (ver. 2.0): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A4, September 2006, accessed December 10, 2012, at http://pubs.water.usgs.gov/twri9A4/

Wilde, F.D., Radtke, D.B., Gibs, Jacob, and Iwatsubo, R.T., eds., 2004 with updates through 2009, Processing of water samples (ver. 2.2): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A5, April 2004, accessed [date viewed], at http://pubs.water.usgs.gov/twri9A5/

Wilde, F.D., ed., 2004, Cleaning of Equipment for water sampling (ver. 2.0): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A3, April 2004, accessed December 10, 2012at http://pubs.water.usgs.gov/twri9A3/

Wilde, F.D., ed., variously dated, Field measurements: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A6, with sec. 6.0–6.8, accessed December 10, 2012 at http://pubs.water.usgs.gov/twri9A6/

Wilde, F.D., 2008, Guidelines for field-measured water-quality properties (ver. 2.0): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A6, section 6.0, accessed December 10, 2012 at http://pubs.water.usgs.gov/twri9A/

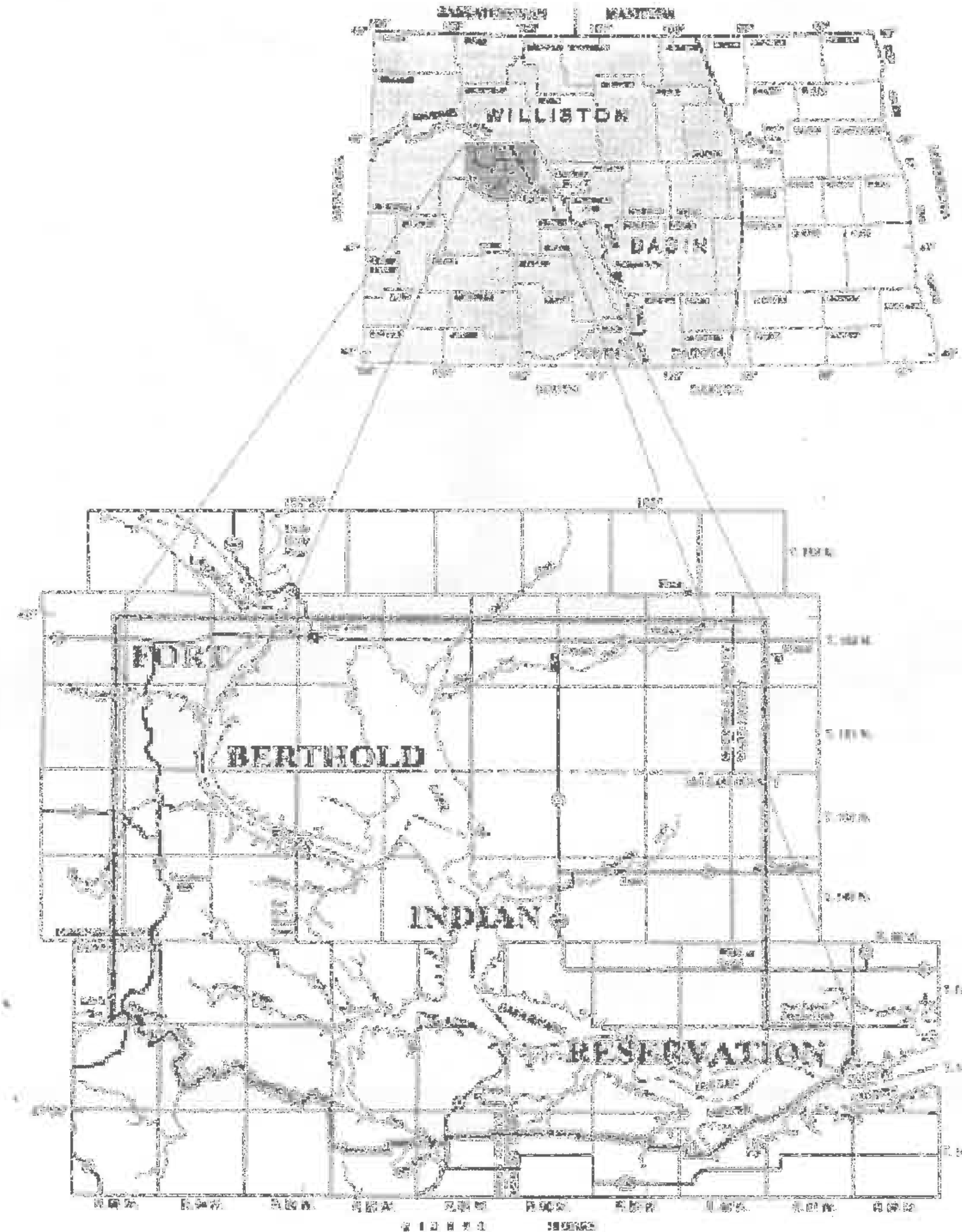


Figure 1. Location of the Fort Berthold Indian Reservation, west-central North Dakota

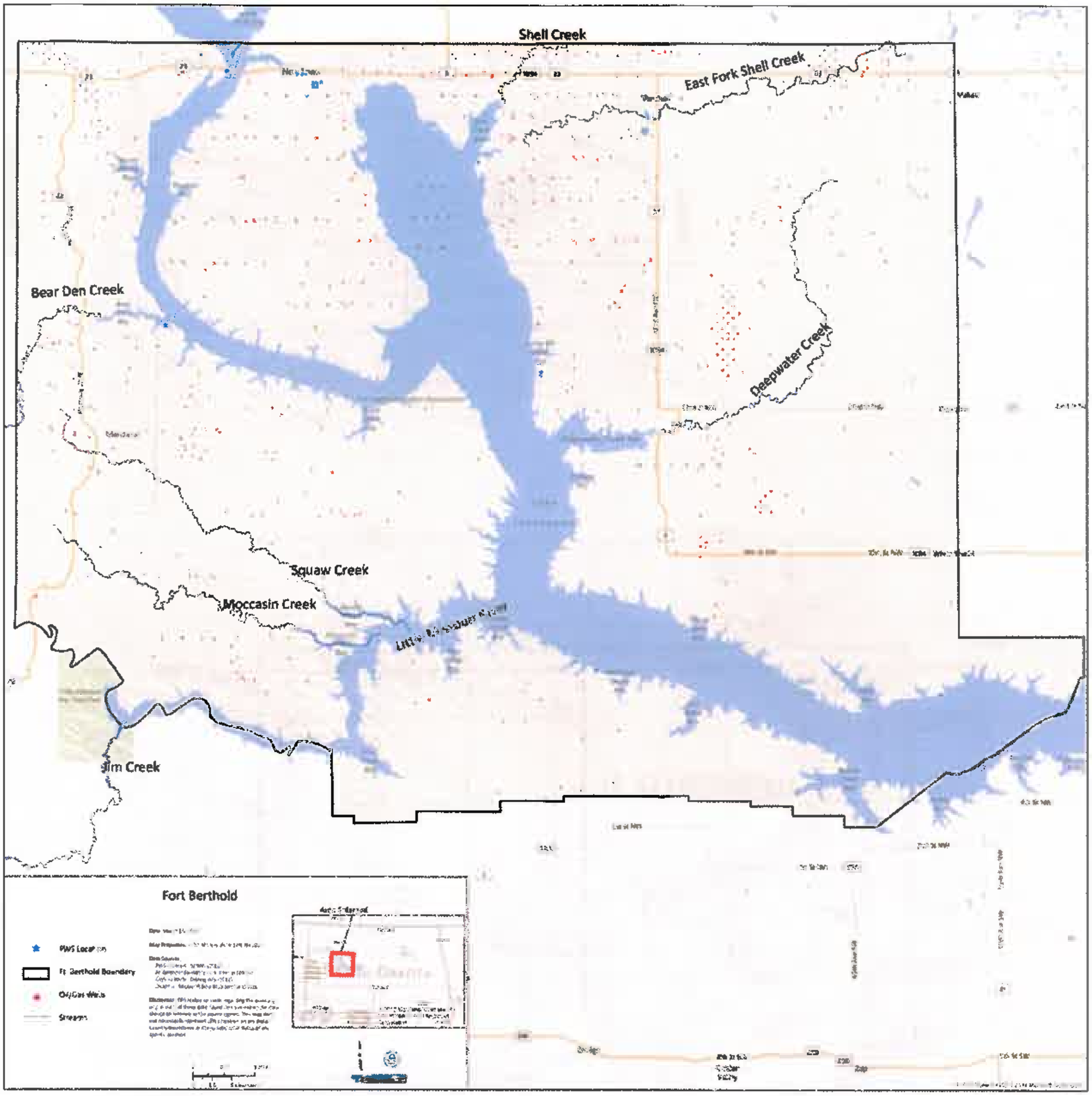


Figure 2. Drainage basins of the six streams to be sampled on the Fort Berthold Indian Reservation

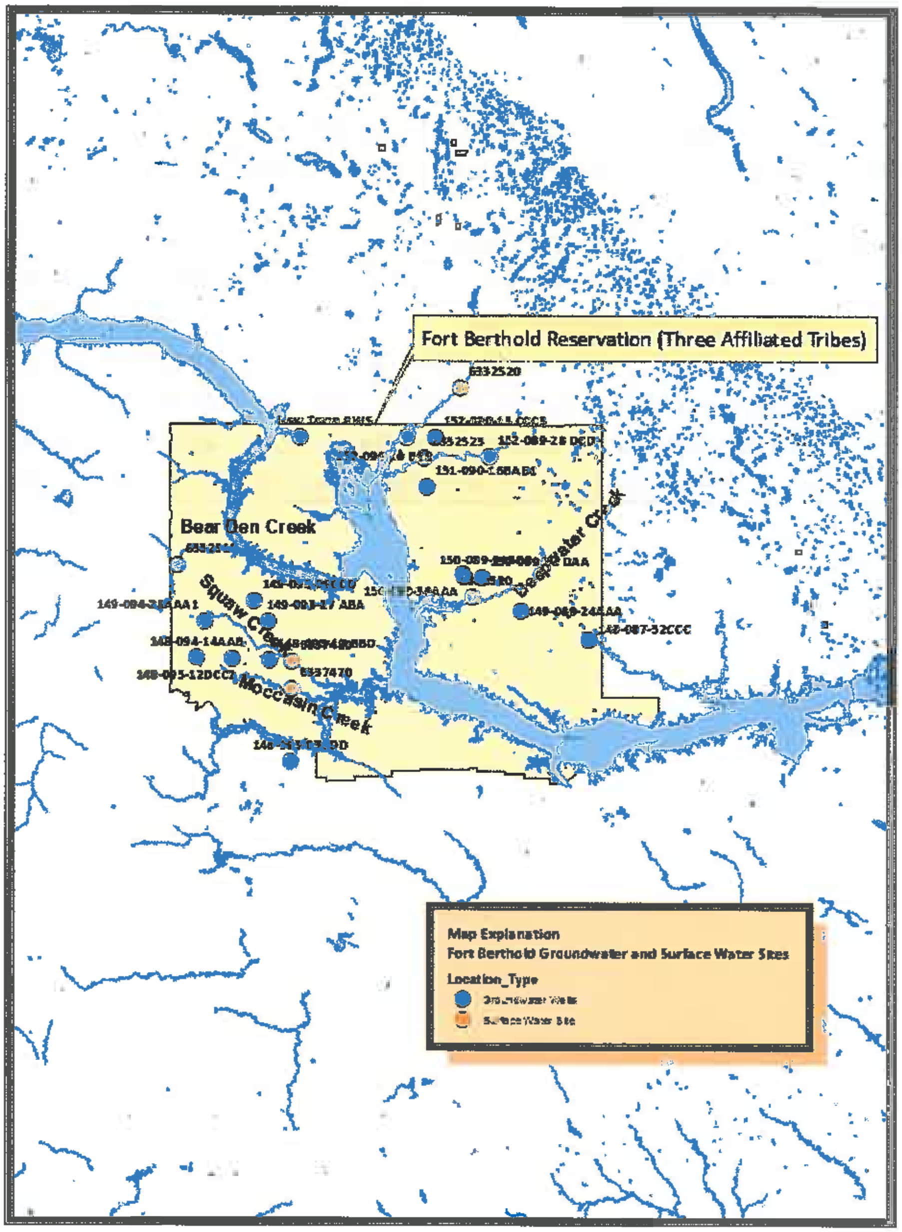
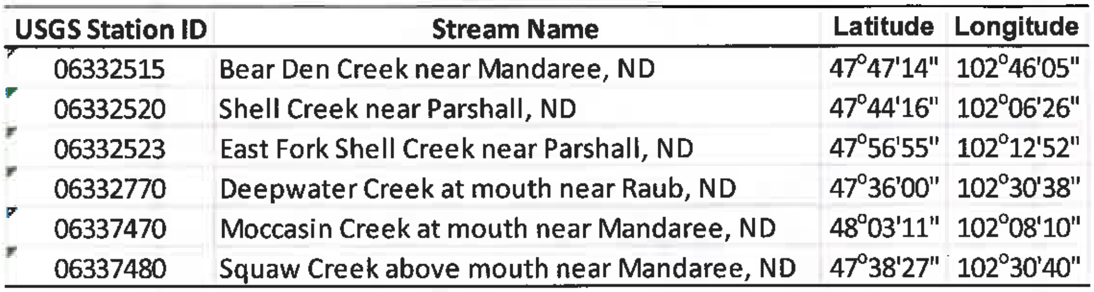


Figure 3. Sampling locations for both surface and groundwater

Table 1. Stream sampling locations
[latitude and longitude coordinates are referenced to the NAD27 datum]



USGS Station ID	Stream Name	Latitude	Longitude
06332515	Bear Den Creek near Mandaree, ND	47°47'14"	102°46'05"
06332520	Shell Creek near Parshall, ND	47°44'16"	102°06'26"
06332523	East Fork Shell Creek near Parshall, ND	47°56'55"	102°12'52"
06332770	Deepwater Creek at mouth near Raub, ND	47°36'00"	102°30'38"
06337470	Moccasin Creek at mouth near Mandaree, ND	48°03'11"	102°08'10"
06337480	Squaw Creek above mouth near Mandaree, ND	47°38'27"	102°30'40"

Table 2. Groundwater sampling locations [latitude and longitude coordinates are referenced to the NAD27 datum]

Local Number	USGS Site ID	Latitude	Longitude	County	Aquifer	Comments
146-093-03CDD	472919102305301	47°29'19"	102°30'53"	Dunn	Fox Hills	This well is 1,525 feet deep and was sampled by Bob Lundgren in August 2013 as part of the USGS Bakken study. May be just outside of the FBIR boundary.
148-093-17 BBD	473834102334301	47°38'34"	102°33'43"	Dunn	Sentinel Butte	This well was located by Greg, Trisha, Dana, and myself in September. It Is on Clem's property. The well depth is 160 feet and we measured a
148-094-14AAB	473840102364501	47°38'40"	102°36'45"	Dunn	Tongue River	Observation well
148-095-12DCC2	473847102432802	47°38'47"	102°43'28"	Dunn	Sentinel Butte	This well is located on the east side of Highway 22. We measured a water level of 15.7 feet from TOC on 9/10/13. This well can easily be
149-087-32CCC	474030101505601	47°40'30"	101°50'56"	McLean	White Shield	observation well on east side; DTW >100 feet; data shows perf'd 338-358
149-089-24AAA	474301102000601	47°43'01"	102°00'06"	McLean	White Shield	observation well on east side; DTW >100 feet; data shows perf'd 158-167
149-093-09CCD	474358102354301	47°43'58"	102°35'43"	Dunn	Sentinel Butte	This well is 65 feet deep and can be sampled. It was located by Trisha and Dana in November per email sent by Trisha on 11/12/13. House well
149-093-27 ABA	474208102334801	47°42'08"	102°33'48"	Dunn	Sentinel Butte	This well is 65 feet deep and can be sampled. It was located by Trisha and Dana in November per email sent by Trisha on 11/12/13. House Well
149-094-28AAA1	474208102422602	47°42'08"	102°42'26"	McKenzie	Sentinel Butte	This well is 120 feet deep and was located by Trisha and Dana in November per email sent by Trisha on 11/12/13. This well was not Included in table 4 of the proposal Bob and I wrote last year. This depth may be the limit to which we can sample. Owner currently has been selling groundwater from an extraction well field. There are two wells at this location, 149-094-AAA2 has been removed for the table--we will only be sampling one of the wells.
150-089-32 DAA	474602102051401	47°46'02"	102°05'14"	McLean	White Shield	This well is 224 feet deep and was located by Trisha and Dana in November per email sent by Trisha on 11/12/13. This well may be too deep for us to sample but it is sampled by the NDSWC. Perhaps we can borrow their equipment? Obeservation well
150-090-36AAA	474628102074901	47°46'28"	102°07'49"	McLean	White Shield	observation well on east side; DTW >100 feet; data shows perf'd 260-300
150-089-31BCC	474608102073901	47°46'08"	102°07'39"	McLean	White Shield	observation well on east side; DTW >100 feet; data shows perf'd 238-278
151-090-16BAB1	475418102123101	47°54'18"	102°12'31"	Mountrail	Sentinel Butte	This well is 190 feet deep and was sampled by Bob Lundgren in August 2013 as part of the USGS Bakken study. This well was not included in my retrospective.
152-089-28 DCD	475700102041401	47°57'00"	102°04'14"	Mountrail	Tongue River	This well is 72 feet deep and can be sampled. It was located by Trisha and Dana in November per email sent by Trisha on 11/12/13. House Well
152-090-15 CCC3	n/a	47°58'43.5"	102°11'31.9"	Mountrail	Shell Creek	Located in same area as Ames Water Depot (goundwater extraction wells for O&G water sells). Ameswater.com 855-776-2694 DTW=45.91' TD=88.96'; data shows perf'd 80-85
152-090-19 BBB	n/a	47°58'39.18"N	102°15'24.34"W	Mountrail	Shell Creek	State Well; DTW=8.7" TD=71.98" no PID/FID detections
New Town PWS	n/a	47°58'40.80"N	102°29'37.30"W		New Town Aquifer	Permission granted to collect samples; contact Steve Ike 701-577-4127
Dakota Plains Water well	n/a	currently being drilled				We have permission to sample new well; they are putting a sampling tap on the well; Keith Wrapp (Pinnacle Engineering) 612-382-3763

Table 3. Field properties and laboratory constituents measured at all sampling locations

Field Parameters			
USGS Parameter Code	**Parameter Name/Description**	**Units**	**Measurement Method**
00400	pH	standard units	YSI Multi-meter 6920 V2-2
00095	Specific Conductance	µS/cm	YSI Multi-meter 6920 V2-2
00010	Water Temperature	°C	YSI Multi-meter 6920 V2-2
00300	Dissolved Oxygen	mg/L	YSI Multi-meter 6920 V2-2
39086	Alkalinity, filtered	mg/L as $CaCO_3$	Field Titration
00453	Bicarbonate, filtered	mg/L	Calculated
00447	Carbonate, filtered	mg/L	Calculated

Table 3. Field properties and laboratory constituents measured at all sampling locations - continued

U.S. EPA, Region 8 Laboratory

4/25/2012

Analytical Method Information

		Reporting	**Surrogate**	**Duplicate**	**Matrix Spike**		**Blank Spike / LCS**	
Analyte	**MDL**	**Limit**	**%R**	**RPD**	**%R**	**RPD**	**%R**	**RPD**
ICP Dissolved Metals -R8 in Water (200.7/6010)								
Preservation: 01-Nitric Acid (HNO3) to pH<2								
Container: 06_125mL Plastic pH <2 w/ HNO3		**Amount Required:** 100ml			**Hold Time:** 180 days			
Silver	2.00	8.00 ug/L		20	80 - 120	20	85 - 115	
Aluminum	25.0	100 ug/L		20	80 - 120	20	85 - 115	
Arsenic	5.00	20.0 ug/L		20	80 - 120	20	85 - 115	
Boron	15.0	100 ug/L		20	80 - 120	20	85 - 115	
Barium	0.800	4.00 ug/L		20	80 - 120	20	85 - 115	
Beryllium	0.200	1.00 ug/L		20	80 - 120	20	85 - 115	
Calcium	10.0	100 ug/L		20	80 - 120	20	85 - 115	
Cadmium	0.200	1.00 ug/L		20	80 - 120	20	85 - 115	
Cobalt	0.300	2.00 ug/L		20	80 - 120	20	85 - 115	
Chromium	0.500	5.00 ug/L		20	80 - 120	20	85 - 115	
Copper	2.00	5.00 ug/L		20	80 - 120	20	85 - 115	
Iron	10.0	100 ug/L		20	80 - 120	20	85 - 115	
Potassium	170	1000 ug/L		20	80 - 120	20	85 - 115	
Magnesium	26.0	100 ug/L		20	80 - 120	20	85 - 115	
Manganese	0.400	2.00 ug/L		20	80 - 120	20	85 - 115	
Molybdenum	1.40	5.00 ug/L		20	80 - 120	20	85 - 115	
Sodium	70.0	500 ug/L		20	80 - 120	20	85 - 115	
Nickel	0.700	4.00 ug/L		20	80 - 120	20	85 - 115	
Lead	3.00	10.0 ug/L		20	80 - 120	20	85 - 115	
Antimony	4.00	20.0 ug/L		20	80 - 120	20	85 - 115	
Selenium	5.00	20.0 ug/L		20	80 - 120	20	85 - 115	
Silica (SiO2)	50.0	200 ug/L		20	80 - 120	20	85 - 115	
Tin	50.0	200 ug/L		20	80 - 120	20	85 - 115	
Strontium	0.400	2.00 ug/L		20	80 - 120	20	85 - 115	
Titanium	2.00	10.0 ug/L		20	80 - 120	20	85 - 115	
Thallium	4.00	20.0 ug/L		20	80 - 120	20	85 - 115	
Vanadium	2.00	10.0 ug/L		20	80 - 120	20	85 - 115	
Zinc	15.0	50.0 ug/L		20	80 - 120	20	85 - 115	
Lithium	11.0	50.0 ug/L		20	80 - 120	20	85 - 115	

Table 3. Field properties and laboratory constituents measured at all sampling locations - continued

U.S. EPA, Region 8 Laboratory

4/25/2012

Analytical Method Information

ICP Total Metals -R8 in Water (200.7/6010)

Preservation: 01-Nitric Acid (HNO3) to pH<2

Container: 06_250mL Plastic pH <2 w/ HNO3 **Amount Required:** 200ml **Hold Time:** 180 days

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Silver	2.00	8.00 ug/L		20	80 - 120	20	85 - 115	
Aluminum	25.0	100 ug/L		20	80 - 120	20	85 - 115	
Arsenic	5.00	20.0 ug/L		20	80 - 120	20	85 - 115	
Barium	0.800	4.00 ug/L		20	80 - 120	20	85 - 115	
Boron	15.0	100 ug/L		20	80 - 120	20	85 - 115	
Beryllium	0.200	1.00 ug/L		20	80 - 120	20	85 - 115	
Calcium	10.0	100 ug/L		20	80 - 120	20	85 - 115	
Cadmium	0.200	1.00 ug/L		20	80 - 120	20	85 - 115	
Cobalt	0.300	2.00 ug/L		20	80 - 120	20	85 - 115	
Chromium	0.500	5.00 ug/L		20	80 - 120	20	85 - 115	
Copper	1.80	5.00 ug/L		20	80 - 120	20	85 - 115	
Iron	10.0	100 ug/L		20	80 - 120	20	85 - 115	
Potassium	170	1000 ug/L		20	80 - 120	20	85 - 115	
Lithium	11.0	50.0 ug/L		20	80 - 120	20	85 - 115	
Magnesium	26.0	100 ug/L		20	80 - 120	20	85 - 115	
Manganese	0.400	2.00 ug/L		20	80 - 120	20	85 - 115	
Molybdenum	1.40	5.00 ug/L		20	80 - 120	20	85 - 115	
Sodium	70.0	500 ug/L		20	80 - 120	20	85 - 115	
Nickel	0.700	4.00 ug/L		20	80 - 120	20	85 - 115	
Lead	3.00	10.0 ug/L		20	80 - 120	20	85 - 115	
Antimony	4.00	20.0 ug/L		20	80 - 120	20	85 - 115	
Selenium	5.00	20.0 ug/L		20	80 - 120	20	85 - 115	
Silica (SiO2)	50.0	200 ug/L		20	80 - 120	20	85 - 115	
Tin	50.0	200 ug/L		20	80 - 120	20	85 - 115	
Strontium	0.400	2.00 ug/L		20	80 - 120	20	85 - 115	
Titanium	1.80	10.0 ug/L		20	80 - 120	20	85 - 115	
Thallium	4.00	20.0 ug/L		20	80 - 120	20	85 - 115	
Vanadium	2.00	10.0 ug/L		20	80 - 120	20	85 - 115	
Zinc	15.0	50.0 ug/L		20	80 - 120	20	85 - 115	

Table 3. Field properties and laboratory constituents measured at all sampling locations - continued

U.S. EPA, Region 8 Laboratory

4/25/2012

Analytical Method Information

ICP-MS dissolved metals -R8 in Water (200.8/6020)

Preservation: 01-Nitric Acid (HNO3) to pH<2

Container: 06_125mL Plastic pH <2 w/ HNO3 **Amount Required:** 100 ml **Hold Time:** 180 days

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike		Blank Spike / LCS	
					%R	RPD	%R	RPD
Beryllium	0.0400	0.200 ug/L		20	80 - 120	20	85 - 115	
Aluminum	14.0	50.0 ug/L		20	80 - 120	20	85 - 115	
Vanadium	1.70	10.0 ug/L		20	80 - 120	20	85 - 115	
Chromium	0.700	5.00 ug/L		20	80 - 120	20	85 - 115	
Manganese	0.200	1.00 ug/L		20	80 - 120	20	85 - 115	
Cobalt	0.0100	0.100 ug/L		20	80 - 120	20	85 - 115	
Nickel	0.160	1.00 ug/L		20	80 - 120	20	85 - 115	
Copper	6.40	20.0 ug/L		20	80 - 120	20	85 - 115	
Zinc	0.700	5.00 ug/L		20	80 - 120	20	85 - 115	
Arsenic	0.440	4.00 ug/L		20	80 - 120	20	85 - 115	
Selenium	0.210	1.00 ug/L		20	80 - 120	20	85 - 115	
Molybdenum	0.0300	0.500 ug/L		20	80 - 120	20	85 - 115	
Silver	0.0300	0.500 ug/L		20	80 - 120	20	85 - 115	
Cadmium	0.0500	0.200 ug/L		20	80 - 120	20	85 - 115	
Antimony	0.100	1.00 ug/L		20	80 - 120	20	85 - 115	
Barium	0.0500	0.300 ug/L		20	80 - 120	20	85 - 115	
Thallium	0.0200	0.300 ug/L		20	80 - 120	20	85 - 115	
Lead	0.0500	1.00 ug/L		20	80 - 120	20	85 - 115	
Thorium	0.0300	0.300 ug/L		20	80 - 120	20	85 - 115	
Uranium	0.0700	0.200 ug/L		20	80 - 120	20	85 - 115	

Table 3. Field properties and laboratory constituents measured at all sampling locations - continued

U.S. EPA, Region 8 Laboratory

4/25/2012

Analytical Method Information

Analyte	**MDL**	**Reporting Limit**	**Surrogate %R**	**Duplicate RPD**	**Matrix Spike**		**Blank Spike / LCS**	
					%R	**RPD**	**%R**	**RPD**
ICP-MS total metals -R8 in Water (200.8/6020)								
Preservation: 01-Nitric Acid (HNO3) to pH<2								
Container: 06_250mL Plastic pH <2 w/ HNO3		**Amount Required:** 100 ml			**Hold Time:** 180 days			
Beryllium	0.0400	0.200 ug/L		20	80 - 120	20	85 - 115	
Aluminum	14.0	50.0 ug/L		20	80 - 120	20	85 - 115	
Vanadium	1.70	10.0 ug/L		20	80 - 120	20	85 - 115	
Chromium	0.700	5.00 ug/L		20	80 - 120	20	85 - 115	
Manganese	0.200	1.00 ug/L		20	80 - 120	20	85 - 115	
Cobalt	0.0100	0.100 ug/L		20	80 - 120	20	85 - 115	
Nickel	0.160	1.00 ug/L		20	80 - 120	20	85 - 115	
Copper	6.40	20.0 ug/L		20	80 - 120	20	85 - 115	
Zinc	0.700	5.00 ug/L		20	80 - 120	20	85 - 115	
Arsenic	0.440	4.00 ug/L		20	80 - 120	20	85 - 115	
Selenium	0.210	1.00 ug/L		20	80 - 120	20	85 - 115	
Molybdenum	0.0300	0.500 ug/L		20	80 - 120	20	85 - 115	
Silver	0.0300	0.500 ug/L		20	80 - 120	20	85 - 115	
Cadmium	0.0500	0.200 ug/L		20	80 - 120	20	85 - 115	
Antimony	0.100	1.00 ug/L		20	80 - 120	20	85 - 115	
Barium	0.0500	0.300 ug/L		20	80 - 120	20	85 - 115	
Thallium	0.0200	0.300 ug/L		20	80 - 120	20	85 - 115	
Lead	0.0500	1.00 ug/L		20	80 - 120	20	85 - 115	
Thorium	0.0200	0.300 ug/L		20	80 - 120	20	85 - 115	
Uranium	0.0700	0.200 ug/L		20	80 - 120	20	85 - 115	

Table 3. Field properties and laboratory constituents measured at all sampling locations - continued

U.S. EPA, Region 8 Laboratory

1/10/2014

Analytical Method Information

Anions by cap IC in Water (EPA 300.0)

Preservation: 07-Ice Only

Container: 03_125mL Plastic Cool to 4° **Amount Required:** 250 mL **Hold Time:** 28 days

C

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Fluoride	0.0100	0.100 mg/L		20	80 - 120	20	85 - 115	20
Chloride	0.0150	0.200 mg/L		20	80 - 120	20	85 - 115	20
Nitrite as N	0.00500	0.0200 mg/L		20	80 - 120	20	85 - 115	20
Sulfate as SO4	0.0700	0.500 mg/L		20	80 - 120	20	85 - 115	20
Bromide	0.0200	0.100 mg/L		20	80 - 120	20	85 - 115	20
Nitrate as N	0.0100	0.0500 mg/L		20	80 - 120	20	85 - 115	20
Orthophosphate as P	0.0100	0.0500 mg/L		20	80 - 120	20	85 - 115	20

U.S. EPA, Region 8 Laboratory

1/10/2014

Analytical Method Information

nutrients - NNP in Water (EPA 365.3)

Preservation: 09-Frozen

Container: 02_60mL Plastic Freeze **Amount Required:** 60 mL **Hold Time:** 28 days

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Orthophosphate as P	2.00	5.00 ug/L		20	80 - 120	20	80 - 120	
Nitrite as N	2.00	5.00 ug/L		20	80 - 120	20	80 - 120	
Nitrate as N	2.00	5.00 ug/L		20	80 - 120	20	80 - 120	

U.S. EPA, Region 8 Laboratory

1/10/2014

Analytical Method Information

Ammonia – R8 in Water (EPA 350.1)

Preservation: 02-Sulfuric Acid (H2SO4) to pH<2, Cool 4°C

Container: 04_125mL Plastic pH <2w/ H2SO4 **Amount Required:** 125mL **Hold Time:** 28 days

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Ammonia as N	0.00400	0.0250 mg/L		20	80 - 120	20	80 - 120	

Table 3. Field properties and laboratory constituents measured at all sampling locations - continued

U.S. EPA, Region 8 Laboratory

1/10/2014

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike		Blank Spike / LCS	
					%R	RPD	%R	RPD
TN by combustion in Water (ASTM 5176) **Preservation:** 04-Hydrochloric Acid (HCl) to pH<2,°Cool 4°C **Container:** 36_125mL Plastic pH<2 w/HCl		**Amount Required:** 125mL			**Hold Time:** 28 days			
Total Nitrogen	0.0300	0.0100 mg/L		20	80 - 120	20	80 - 120	

U.S. EPA, Region 8 Laboratory

1/10/2014

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike		Blank Spike / LCS	
					%R	RPD	%R	RPD
TP R8 in Water (EPA 365.4 - TP) **Preservation:** 02-Sufuric Acid (H2SO4) to pH<2, Cool 4 °C **Container:** 04_125mL Plastic pH<2 w/H2SO4		**Amount Required:** 125mL			**Hold Time:** 28 days			
Phosphorus	0.00400	0.0100 mg/L		20	80 - 120	20	80 - 120	

Table 3. Field properties and laboratory constituents measured at all sampling locations – continued for Frac Schedules 8260 and 8270

U.S. EPA, Region 8 Laboratory

5/14/2013

Analytical Method Information

8260 Frac 2013 in Water (EPA 8260B)

Preservation: 07-Ice Only

Container: 00_40mL Clear Vial Cool to 4° C **Amount Required:** 40ml **Hold Time:** 14 days

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Fluorobenzene	0.00							
Dichlorodifluoromethane	0.287	1.00 ug/L		20	30 - 155	20	30 - 155	
Chloromethane	0.250	0.250 ug/L		20	40 - 125	20	40 - 125	
Vinyl chloride	0.0656	0.250 ug/L		20	50 - 145	20	50 - 145	
Bromomethane	0.250	0.250 ug/L		20	30 - 145	20	30 - 145	
Chloroethane	0.0520	0.250 ug/L		20	60 - 135	20	60 - 135	
Trichlorofluoromethane	0.0520	0.250 ug/L		20	60 - 145	20	60 - 145	
Ethyl Ether	0.0360	0.250 ug/L		20	50 - 130	20	50 - 130	
1,1-Dichloroethene	0.0640	0.250 ug/L		20	70 - 130	20	70 - 130	
Carbon disulfide	0.0393	0.250 ug/L		20	35 - 160	20	35 - 160	
Iodomethane	0.0450	0.250 ug/L		20	50 - 130	20	50 - 130	
Allyl chloride	0.0994	0.250 ug/L		20	50 - 130	20	50 - 130	
Acetone	0.138	1.00 ug/L		20	40 - 140	20	40 - 140	
Methylene chloride	0.0453	0.250 ug/L		20	55 - 140	20	55 - 140	
trans-1,2-Dichloroethene	0.0661	0.250 ug/L		20	60 - 140	20	60 - 140	
Methyl tert-Butyl Ether	0.0410	0.250 ug/L		20	65 - 125	20	65 - 125	
1,1-Dichloroethane	0.0310	0.250 ug/L		20	70 - 135	20	70 - 135	
Acrylonitrile	0.0393	0.250 ug/L		20	50 - 130	20	50 - 130	
cis-1,2-Dichloroethene	0.0800	0.250 ug/L		20	70 - 125	20	70 - 125	
2,2-Dichloropropane	0.0650	0.250 ug/L		20	70 - 135	20	70 - 135	
Bromochloromethane	0.0297	0.250 ug/L		20	65 - 130	20	65 - 130	
Chloroform	0.0297	0.250 ug/L		20	65 - 135	20	65 - 135	
Methyl Acrylate	0.0280	0.250 ug/L		20	50 - 130	20	50 - 130	
surr: Dibromofluoromethane			85 - 115	20				
Carbon tetrachloride	0.0636	0.250 ug/L		20	65 - 140	20	65 - 140	
2-Butanone	0.124	0.500 ug/L		20	30 - 150	20	30 - 150	
1,1,1-Trichloroethane	0.0560	0.250 ug/L		20	65 - 130	20	65 - 130	
1,1-Dichloropropene	0.0921	0.250 ug/L		20	75 - 130	20	75 - 130	
surr: 1,2-Dichloroethane-d4			70 - 120	20		20		
Benzene	0.0578	0.250 ug/L		20	80 - 120	20	80 - 120	
Methacrylonitrile	0.0374	0.250 ug/L		20	50 - 130	20	50 - 130	
1,2-Dichloroethane	0.0358	0.250 ug/L		20	70 - 130	20	70 - 130	
Trichloroethene	0.0578	0.250 ug/L		20	70 - 125	20	70 - 125	
Dibromomethane	0.0580	0.250 ug/L		20	75 - 125	20	75 - 125	
1,2-Dichloropropane	0.0280	0.250 ug/L		20	75 - 125	20	75 - 125	
Bromodichloromethane	0.0250	0.250 ug/L		20	75 - 120	20	75 - 120	
Chlorobenzene-d5								
cis-1,3-Dichloropropene	0.0210	0.250 ug/L		20	70 - 130	20	70 - 130	
surr: Toluene-d8			85 - 120	20				
Toluene	0.0437	0.250 ug/L		20	75 - 120	20	75 - 120	
4-Methyl-2-pentanone	0.0318	0.250 ug/L		20	60 - 135	20	60 - 135	
trans-1,3-Dichloropropene	0.0275	0.250 ug/L		20	55 - 140	20	55 - 140	
Tetrachloroethene	0.216	0.250 ug/L		20	45 - 150	20	45 - 150	
1,1,2-Trichloroethane	0.0349	0.250 ug/L		20	75 - 125	20	75 - 125	
Chlorodibromomethane	0.0360	0.250 ug/L		20	60 - 135	20	60 - 135	
1,3-Dichloropropane	0.0160	0.250 ug/L		20	75 - 125	20	75 - 125	

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike		Blank Spike / LCS	
					%R	RPD	%R	RPD
1,2-Dibromoethane (EDB)	0.0470	0.250 ug/L		20	80 - 120	20	80 - 120	
2-Hexanone	0.0785	0.250 ug/L		20	55 - 130	20	55 - 130	
Chlorobenzene	0.0227	0.250 ug/L		20	80 - 120	20	80 - 120	
Ethylbenzene	0.0310	0.250 ug/L		20	75 - 125	20	75 - 125	
1,1,1,2-Tetrachloroethane	0.0360	0.250 ug/L		20	80 - 130	20	80 - 130	
m,p-Xylene	0.0780	0.500 ug/L		20	75 - 125	20	75 - 125	
o-Xylene	0.0675	0.250 ug/L		20	80 - 125	20	80 - 125	
Styrene	0.0422	0.250 ug/L		20	65 - 135	20	65 - 135	
Bromoform	0.0594	0.250 ug/L		20	70 - 130	20	70 - 130	
Isopropylbenzene	0.0717	0.250 ug/L		20	75 - 125	20	75 - 125	
1,4-Dichlorobenzene-d4								
surr: 4-Bromofluorobenzene			75 - 120	20				
Bromobenzene	0.0797	0.250 ug/L		20	75 - 125	20	75 - 125	
n-Propyl Benzene	0.0250	0.250 ug/L		20	70 - 130	20	70 - 130	
1,1,2,2-Tetrachloroethane	0.181	0.250 ug/L		20	65 - 130	20	65 - 130	
2-Chlorotoluene	0.0903	0.250 ug/L		20	75 - 125	20	75 - 125	
1,3,5-Trimethylbenzene	0.0751	0.250 ug/L		20	75 - 130	20	75 - 130	
1,2,3-Trichloropropane	0.0416	0.250 ug/L		20	75 - 125	20	75 - 125	
4-Chlorotoluene	0.0360	0.250 ug/L		20	75 - 130	20	75 - 130	
tert-Butylbenzene	0.0390	0.250 ug/L		20	70 - 130	20	70 - 130	
1,2,4-Trimethylbenzene	0.0320	0.250 ug/L		20	75 - 130	20	75 - 130	
sec-Butylbenzene	0.0360	0.250 ug/L		20	70 - 125	20	70 - 125	
p-Isopropyltoluene	0.0759	0.250 ug/L		20	75 - 130	20	75 - 130	
1,3-Dichlorobenzene	0.0360	0.250 ug/L		20	75 - 125	20	75 - 125	
1,4-Dichlorobenzene	0.0280	0.250 ug/L		20	75 - 125	20	75 - 125	
n-Butyl Benzene	0.0360	0.250 ug/L		20	70 - 135	20	70 - 135	
1,2-Dichlorobenzene	0.0280	0.250 ug/L		20	70 - 120	20	70 - 120	
Adamantane	0.0759	0.250 ug/L		20	70 - 130	20	70 - 130	
1,3-Dimethyl adamantane	0.0530	0.250 ug/L		20	70 - 130	20	70 - 130	
1,2-Dibromo-3-chloropropane	0.201	0.250 ug/L		20	50 - 130	20	50 - 130	
Hexachlorobutadiene	0.0507	0.250 ug/L		20	50 - 140	20	50 - 140	
1,2,4-Trichlorobenzene	0.0220	0.250 ug/L		20	65 - 135	20	65 - 135	
Naphthalene	0.0424	0.250 ug/L		20	55 - 140	20	55 - 140	
1,2,3-Trichlorobenzene	0.0350	0.250 ug/L		20	55 - 140	20	55 - 140	
Xylenes (total)	1.00	1.00 ug/L		20	75 - 125	20	75 - 125	
Tentatively Identified Compounds								

U.S. EPA, Region 8 Laboratory

5/1/2013

Analytical Method Information

8270 Frac Full in Water (EPA 8270D)

Preservation: 07-Ice Only

Container: 12_1000mL Amber Glass pH <2 w/ HCl **Amount Required:** 1L **Hold Time:** 7 days

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
1,4-Dichlorobenzene-d4								
N-Nitrosodimethylamine	0.488	1.00 ug/L		20	25 - 110	30	25 - 110	30
Pyridine	0.0140	1.00 ug/L		20	0 - 150	30	0 - 150	30
surr: 2-Fluorophenol			20 - 110					
2-Butoxyethanol	0.698	1.00 ug/L		20	60 - 130	30	60 - 130	30
Aniline	0.202	1.00 ug/L		20	0 - 150	30	0 - 110	20
Bis(2-chloroethyl)ether	0.463	1.00 ug/L		20	35 - 110	30	35 - 110	20
surr: Phenol-d6			10 - 115					
Phenol	0.967	2.00 ug/L		20	20 - 115	30	20 - 115	20
2-Chlorophenol	0.911	2.00 ug/L		20	35 - 105	30	35 - 105	20
1,3-Dichlorobenzene	0.375	1.00 ug/L		20	30 - 100	30	30 - 100	20
1,4-Dichlorobenzene	0.377	1.00 ug/L		20	30 - 100	30	30 - 100	20
(R)-(+)-Limonene	0.257	1.00 ug/L		20	60 - 130	30	60 - 130	30
1,2-Dichlorobenzene	0.399	1.00 ug/L		20	35 - 100	30	35 - 100	20
Benzyl alcohol	0.549	1.00 ug/L		20	50 - 150	30	50 - 150	20
Bis(2-chloroisopropyl)ether	0.480	1.00 ug/L		20	25 - 130	30	25 - 130	20
2-Methylphenol	0.999	2.00 ug/L		20	40 - 110	30	40 - 110	20
N-Nitrosodi-n-propylamine	0.598	1.00 ug/L		20	35 - 130	30	35 - 130	20
Adamantane	0.280	1.00 ug/L		20	60 - 130	30	60 - 130	30
Naphthalene-d8								
surr: Nitrobenzene-d5			40 - 110					
Nitrobenzene	0.453	1.00 ug/L		20	45 - 110	30	45 - 110	20
Hexachloroethane	0.320	1.00 ug/L		20	30 - 95	30	30 - 95	20
3 & 4-Methylphenol	2.08	5.00 ug/L		20	30 - 110	30	30 - 110	20
Isophorone	0.578	1.00 ug/L		20	50 - 110	30	50 - 110	20
1,3-Dimethyl adamantane	0.277	1.00 ug/L		20	60 - 130	30	60 - 130	30
2-Nitrophenol	0.864	2.00 ug/L		20	40 - 115	30	40 - 115	20
Bis(2-chloroethoxy)methane	0.523	1.00 ug/L		20	45 - 105	30	45 - 105	20
2,4-Dimethylphenol	0.937	2.00 ug/L		20	30 - 110	30	30 - 110	20
2,4-Dichlorophenol	1.05	2.00 ug/L		20	50 - 105	30	50 - 105	20
Benzoic acid	1.59	3.00 ug/L		20	20 - 115	30	20 - 115	20
1,2,4-Trichlorobenzene	0.399	1.00 ug/L		20	35 - 105	30	35 - 105	20
Naphthalene	0.426	1.00 ug/L		20	40 - 100	30	40 - 100	20
Terpiniol	0.617	1.00 ug/L		20	60 - 130	30	60 - 130	30
4-Chloroaniline	1.05	3.00 ug/L		20	15 - 110	30	15 - 110	20
Hexachlorobutadiene	0.304	1.00 ug/L		20	25 - 105	30	25 - 105	20
2-Methylnaphthalene	0.468	1.00 ug/L		20	45 - 105	30	45 - 105	20
1-Methylnaphthalene	0.482	1.00 ug/L		20	45 - 105	30	45 - 105	20
4-Chloro-3-methylphenol	1.22	2.00 ug/L		20	45 - 110	30	45 - 110	20
Hexachlorocyclopentadiene	0.227	1.00 ug/L		20	0 - 95	30	0 - 95	20
2,4,6-Trichlorophenol	1.19	2.00 ug/L		20	50 - 115	30	50 - 115	20
2,4,5-Trichlorophenol	1.15	2.00 ug/L		20	50 - 110	30	50 - 110	20
surr: 2-Fluorobiphenyl			50 - 110					
2-Chloronaphthalene	0.498	1.00 ug/L		20	50 - 105	30	50 - 105	20
2-Nitroaniline	0.556	1.00 ug/L		20	50 - 115	30	50 - 115	20
1,4-Dinitrobenzene	0.450	1.00 ug/L		20	45 - 110	30	45 - 110	20

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike		Blank Spike / LCS	
					%R	RPD	%R	RPD
1,3-Dinitrobenzene	0.460	1.00 ug/L		20	45 - 110	30	45 - 110	20
2,6-Dinitrotoluene	0.497	1.00 ug/L		20	50 - 115	30	50 - 115	20
Acenaphthylene	0.562	1.00 ug/L		20	50 - 105	30	50 - 105	20
1,2-Dinitrobenzene	0.460	1.00 ug/L		20	45 - 110	30	45 - 110	20
Dimethyl phthalate	0.516	1.00 ug/L		20	25 - 125	30	25 - 125	20
Acenaphthene-d10								
3-Nitroaniline	1.30	3.00 ug/L		20	20 - 125	30	20 - 125	20
2,4-Dinitrophenol	1.75	3.00 ug/L		20	15 - 140	30	15 - 140	20
Acenaphthene	0.588	1.00 ug/L		20	45 - 110	30	45 - 110	20
2,4-Dinitrotoluene	0.413	1.00 ug/L		20	50 - 120	30	50 - 120	20
Dibenzofuran	0.589	1.00 ug/L		20	55 - 105	30	55 - 105	20
4-Nitrophenol	1.08	3.00 ug/L		20	0 - 125	30	0 - 125	20
2,3,5,6-Tetrachlorophenol	1.05	2.00 ug/L		20	50 - 110	30	50 - 110	20
2,3,4,6-Tetrachlorophenol	1.08	2.00 ug/L		20	50 - 110	30	50 - 110	20
Fluorene	0.626	1.00 ug/L		20	50 - 110	30	50 - 110	20
4,6-Dinitro-2-methylphenol	0.958	2.00 ug/L		20	40 - 130	30	40 - 130	20
Diethyl phthalate	0.480	1.00 ug/L		20	40 - 120	30	40 - 120	20
4-Chlorophenyl phenyl ether	0.612	1.00 ug/L		20	50 - 110	30	50 - 110	20
4-Nitroaniline	1.13	3.00 ug/L		20	35 - 120	30	35 - 120	20
Diphenylamine	0.521	1.00 ug/L		20	55 - 115	30	55 - 115	20
Azobenzene	0.596	1.00 ug/L		20	50 - 115	30	50 - 115	20
surr: 2,4,6-Tribromophenol			40 - 125					
4-Bromophenyl phenyl ether	0.566	1.00 ug/L		20	50 - 115	30	50 - 115	20
Hexachlorobenzene	0.487	1.00 ug/L		20	50 - 110	30	50 - 110	20
Pentachlorophenol	0.928	2.00 ug/L		20	40 - 115	30	40 - 115	20
Phenanthrene-d10								
Phenanthrene	0.411	1.00 ug/L		20	50 - 115	30	50 - 115	20
Anthracene	0.410	1.00 ug/L		20	55 - 110	30	55 - 110	20
Carbazole	0.913	3.00 ug/L		20	50 - 115	30	50 - 115	20
Di-n-butyl phthalate	0.626	1.00 ug/L		20	55 - 115	30	55 - 115	20
Fluoranthene	0.384	1.00 ug/L		20	55 - 115	30	55 - 115	20
Pyrene	0.386	1.00 ug/L		20	50 - 130	30	50 - 130	20
surr: Terphenyl-d14			50 - 135					
Butyl benzyl phthalate	0.610	1.00 ug/L		20	45 - 115	30	45 - 115	20
Chrysene-d12								
Bis-(2-Ethylhexyl) Adipate	0.494	1.00 ug/L		20	40 - 125	30	40 - 125	20
2-Butoxyethanol phosphate	0.698	1.00 ug/L		20	60 - 130	30	60 - 130	30
Benzo (a) anthracene	0.377	1.00 ug/L		20	55 - 110	30	55 - 110	20
3,3′-Dichlorobenzidine	0.558	1.00 ug/L		20	0 - 150	30	0 - 150	20
Chrysene	0.340	1.00 ug/L		20	55 - 110	30	55 - 110	20
Bis(2-ethylhexyl)phthalate	1.12	2.00 ug/L		20	40 - 125	30	40 - 125	20
Di-n-octyl phthalate	0.544	1.00 ug/L		20	35 - 135	30	35 - 135	20
Benzo (b) fluoranthene	0.428	1.00 ug/L		20	45 - 120	30	45 - 120	20
Benzo (k) fluoranthene	0.416	1.00 ug/L		20	45 - 125	30	45 - 125	20
Squalene	1.33	2.00 ug/L		20	60 - 130	30	60 - 130	30
Benzo (a) pyrene	0.475	1.00 ug/L		20	55 - 110	30	55 - 110	20
Perylene-d12								
Inderio (1,2,3-cd) pyrene	0.441	1.00 ug/L		20	45 - 125	30	45 - 125	20
Dibenz (a,h) anthracene	0.425	1.00 ug/L		20	40 - 125	30	40 - 125	20
Benzo (g,h,i) perylene	0.423	1.00 ug/L		20	40 - 125	30	40 - 125	20

Appendix I – EPA Region 8 Laboratory QA Manual

U.S. EPA Region 8 Environmental Laboratory

Quality Assurance Manual

SOP No.: QSP-001 Rev. 1.2

Effective Date: June 9, 2011

Mark Burkhardt, PhD, Laboratory Director
William Batschelet, PhD, Quality Assurance Officer

16194 West 45th Drive
Golden, CO 80403
Phone: 303-312-7700

This page is left blank intentionally

EPA Region 8 Laboratory
16194 West 45th Drive
Golden, CO 80403
303-312-7700

ADMINISTRATIVE STANDARD OPERATING PROCEDURE

SOP No.: QSP-001 Rev.: 1.2

TITLE: **U.S. EPA Region 8 Environmental Laboratory Quality Assurance Manual**

Dept./Position	**Name**	**Signature**	**Date Reviewed/ Approved**
Region 8 Lab QA Officer	William Batschelet, PhD		9 Jun 11
Region 8 Lab Director	Mark Burkhardt, PhD		6/9/11

Effective Date: 6/9/11

Revision/Change History

Revision	Change Description	Effective Date
1.0	Assigned new SOP number QSP-001 Rev. 1.0. Updated format. Updated for current processes and procedures.	11/3/10
1.1	Minor editorial changes. Updates to Section 4.2.2 (IDC), Section 4.8 (Handling of Samples), and Appendix A (Analytical Capabilities).	05/02/11
1.2	Minor editorial changes. Update to Section 3.13 regarding records retention and disposition.	06/09/11

Table of Contents

1. INTRODUCTION

The U.S. Environmental Protection Agency (EPA) implements Federal laws designed to promote public health by protecting our Nation's air, water, and soil from harmful pollution. EPA accomplishes its mission by a variety of research, monitoring, standard setting, and enforcement activities. EPA also coordinates and supports research and antipollution activities of State and local and tribal governments, private and public groups, individuals, and educational institutions.

1.1. Scope

The Region 8 Laboratory (R8L) provides science support and environmental analytical services for these Regional Programs. It provides analytical testing for water and solid samples, hazardous wastes, as well as microbiological and biological testing. The laboratory also provides technical consulting services such as development and review of sampling and quality assurance plans, interpretation of chemistry data, and implementation of procedures and training for sampling programs. The environmental analytical services provided by the Laboratory are conducted and completed within critical time frames for use in deliberations and decision-making in various Region 8 and National EPA programs. The scientific expertise required to design and execute these analyses is also called upon to provide advice and expert testimony in Regional and National Program decisions. Appendix A contains a listing of R8L analytical capabilities.

1.2. Mission

The R8L provides the EPA and the public with high quality, reliable consulting, and field sampling, biological, and analytical data to describe and understand the environment through the impartial application of science and technology to physical, chemical, and biological analyses of water.

1.3. Purpose

The purpose of this Quality Assurance Manual (QAM) is to outline the quality system for the laboratory. The QAM defines the policies, procedures, and documentation that assure analytical services continually meet a defined standard of quality that is designed to provide clients with data of known and documented quality and, where applicable, demonstrate regulatory compliance. It sets the standard under which all laboratory operations are performed including the laboratory's organization, objectives, and operating philosophy.

2. QUALITY POLICY STATEMENT

It is the policy of the R8L that all data generated by the Laboratory are of the highest quality and meet or exceed the data quality objectives of each project. Management and analytical staff of share the responsibility of insuring that the data produced is scientifically sound and well documented. It is of utmost importance that the quality of all data produced by the R8L be well defined and communicated to the end user(s) of the data. This policy is implemented by:

- having in place and following a complete and systematic process of quality control activities to assist in defining data quality;
- insuring that data quality is documented and communicated to all users of the data by assigning appropriate qualifier flags according to prescribed procedures; and
- having a cross-check system review process to verify that data are generated in accordance with sound and appropriate technical procedures and insuring that all activities associated with the analyses, calculations and data reduction are complete and accurate.

The R8L governing principles are as follows:

1. To continually improve service for consulting, field analysis, analytical chemistry, and biology,
2. To reduce chemical waste and improve waste management, and
3. To maintain the highest quality possible in all aspects of its work.

These governing principles require that new method development incorporate the best science and technology. All new methods or equipment also must reduce chemical waste for environmental protection, for operational efficiency, and for financial reasons.

The R8L staff are dedicated, experienced professionals and are expected to maintain personal accountability for the quality of their work. They are expected to conduct themselves with the highest regard for ethics and for data integrity. Each person involved with the laboratory's operations shall be familiar with the overall function of the quality system and the specific application of the quality system to the tests that they conduct and activities they perform. Analytical services are expected to be impartial, consistent and first-rate. Implemented technology is continually improved and upgraded to meet analytical requirements. The R8L must consistently produce consulting, field, and analytical data of the highest quality that meets the needs of the EPA.

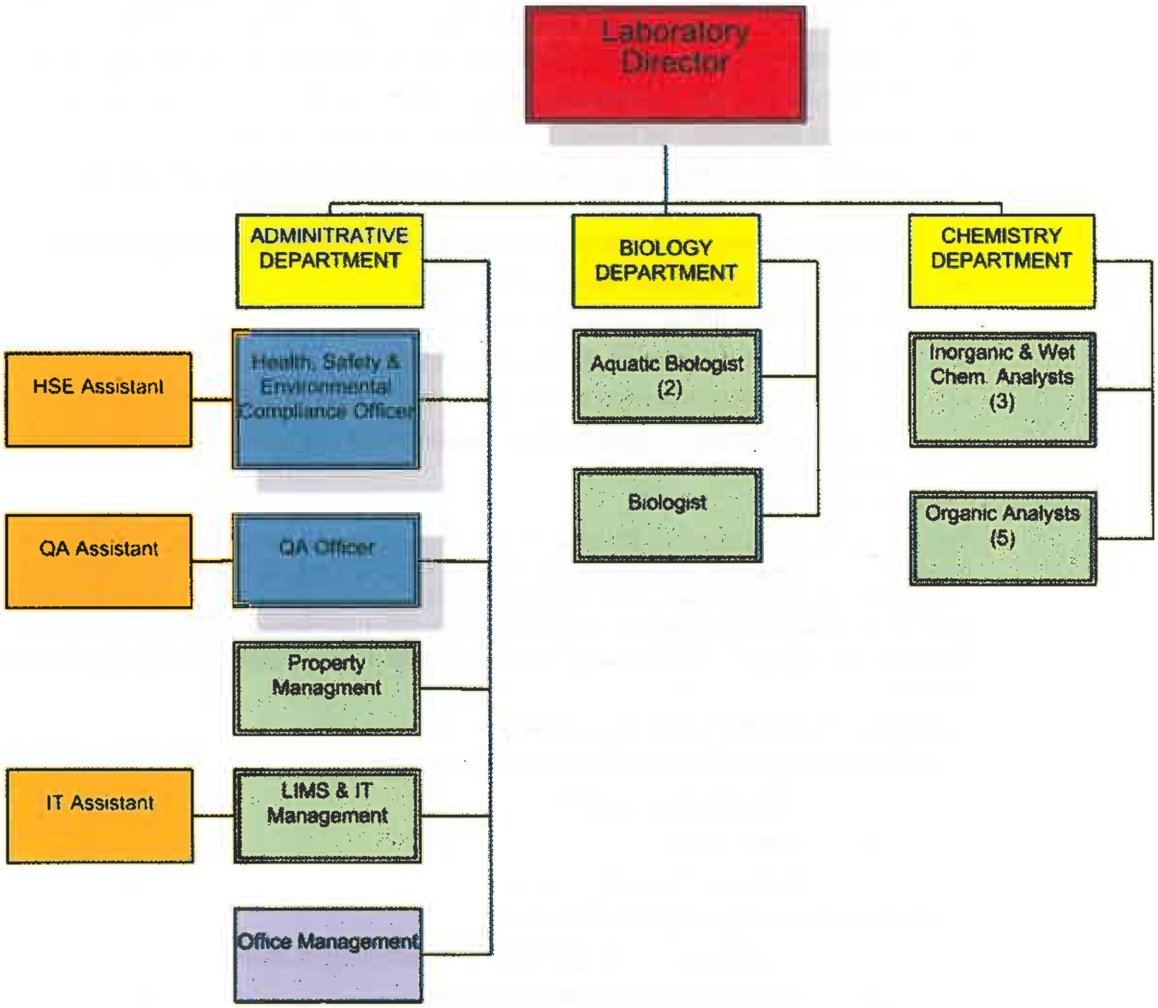
All employees are trained annually on ethical principles and procedures surrounding the data that is generated.

3. MANAGEMENT REQUIREMENTS

3.1. Organization

The R8L is part of Technical and Management Services Program Office (TMS) within EPA Region 8. It is a Regional asset and is the principal analytical laboratory serving the other program offices within the Region.

Region 8 Laboratory



3.2. Personnel Responsibilities

3.2.1. Laboratory Director

The R8L Director is responsible overall operation and management of the laboratory and for ensuring its success. That responsibility includes oversight for issuing, implementing, and interpreting policy guidelines. The guidelines are meant principally to provide support to the operation of the R8L in carrying out its mission. The support functions include management of the R8L, administrative support, and safety compliance for the health and well-being of all R8L scientists and staff.

The Director is responsible for carrying out the directives of the Region and for ensuring staff, under the direction of the Director, follow those directives. The Director reports directly to the Assistant Regional Administrator of TMS.

3.2.2. Laboratory Quality Assurance Officer

The LQAO is responsible for monitoring compliance of the Region 8 Laboratory with national EPA Quality Assurance (QA) and Quality Control (QC) directives and guidance, and with the Region 8 Laboratory QA and QC policies and procedures. The LQAO reports directly to the Laboratory Director. In some cases, the LQAO, with approval from the Laboratory Director, delegates the workload associated with a specific responsibility. In those situations, it is the responsibility of the LQAO to maintain oversight of delegated activities. Specific responsibilities include, but are not limited to, the following:

- Serve as the focal point for QA and QC activities within the Region 8 Laboratory,
- Perform QC review of laboratory reports, including issuing the final reports,
- Maintain and revise, as needed, the Quality Assurance Manual,
- Maintain and initiate review, as needed, of Standard Operating Procedures (SOPs),
- Maintain the results of and the scheduling of Performance Testing Samples,
- Provide oversight and/or review of QC data,
- Monitor and/or initiate corrective actions,
- Conduct internal audits on the technical operation annually,
- Notify Laboratory management of deficiencies in the quality system, and
- Provide quality documentation and assistance as requested to the Region 8 Quality Assurance Manager.

3.2.3. Laboratory Staff

The laboratory is currently staffed by 20 full-time positions. All laboratory personnel involved in the analysis, interpretation, and approval of data have the responsibility to understand and follow the QAM. Detailed qualifications and training summaries for each staff member will be maintained according the SOP "Laboratory Training" and will be available on request. The following are the general responsibilities associated with each position:

Laboratory Administration: 5 positions

Office Manager / Physical Science Technician

- Administrative support
- Timekeeping
- Maintain chemical inventory

Field / Laboratory Property Specialist

- Inventory and maintain field and laboratory supplies
- Order field and laboratory supplies
- Pack and ship field sampling supplies and containers

Health and Safety / Environmental Compliance Officer: 2 positions

- Responsible for the facilities management
- Responsible for laboratory security
- Responsible for environmental compliance
- Responsible for occupational health and safety
- Responsible for pollution prevention

Quality Assurance Assistant (QAA)

- Provide support to the LQAO
- Provide document management support
- Assist with training
- Assist with tracking QA goals and requirements

LIMS Administrator / Computer Specialist: 2 positions

- Manage the Laboratory Information Management System (LIMS)
- Manage the laboratory chemical inventory system
- Oversee and resolve hardware, software, and network issues for laboratory equipment

Analytical Chemists: 8 positions

- Analyze and report the results of chemical analysis of environmental samples for the Regional laboratory
- Responsible for method development and validation, data quality checks, and operation and maintenance of analytical instruments
- Recommend procedures and coordinate technical activities within the laboratory
- Train, review, and certify performance of analysts during training activities
- Serve as technical adviser to clients and as team leader for more complex projects
- Responsible for technical procurement and specifications for new instruments and equipment

Biologists / Microbiologist: 3 positions

- Analyze and report the results of microbiological analysis of environmental samples for the Regional laboratory
- Perform special studies in microbiology for the Region
- Advise and assist in field operations and sampling

3.3. Quality System

The laboratory's Quality System is documented in this QAM and associated quality system documents. Together they describe the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of the organization for ensuring quality in its work processes, products, and services.

3.3.1. Quality Assurance Manual

The most current version of the QAM is maintained both in hard copy and electronically by the Laboratory Quality Assurance Officer (LQAO). The manual is available to all EPA staff in a read-only version on the Laboratory's G: drive at G:\Released Documents\ QAM. While hard copies of the manual may be printed, it is the responsibility of each individual to ensure that they are using the most current version.

The manual will be reviewed annually by the LQAO and Laboratory Director. If no changes are made, the date of annual review will be listed in the Revision/Change History section.

In an effort to keep the manual as up-to-date as possible, changes may be made as part of the annual review or at any time deemed appropriate during the calendar year. When such a change is made, the changes will be listed in the Revision/Change History section of the document, and a new revision number as outlined in the *Controlled Document Number Assignment Procedure* (QSP-002) will be assigned. The document will then go through the review and approval process described in Section 3.4.

3.4. Document Control

The R8L has established procedures to control all documents that form part of its quality system. This is outlined in *Controlled Document Review, Approval, Change, and Obsolete Procedure* (QSP-003). All documents issued to personnel in the Laboratory as part of its quality system are reviewed and approved for use by the Laboratory Director and LQAO prior to release. The most current versions of controlled documents are maintained both in hard copy and electronically by the LQAO, and read-only versions of approved documents are available to all personnel on the Laboratory's G: drive at G:\Lab QA\Released Documents\. Current editable document files are located in a limited access location on the Lab Network drive.

All obsolete and changed electronic document files are located in a limited access location on the Lab Network drive. All obsolete and changed hard copy original documents are filed in an appropriate document obsolete file. These are maintained for a period of five years. After that period, they shall be transferred to the EPA central archives.

3.4.1. Standard Operating Procedures (SOPs)

Laboratory SOPs are developed using a peer review process with final review and approval by the LQAO and Laboratory Director. The process is outlined in the Standard Operating Procedure Writing, Content, and Formatting Procedure (QSP-800), which is consistent with the guidelines established in U.S. EPA Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality-Related Documents, EPA QA/G-6, April 2007. SOP types are Administrative, Technical, or Equipment, and the content for each is outlined.

SOPs must be reviewed periodically to ensure that they reflect current Laboratory activities. This review should occur at least every two years. If an analytical method changes, the SOP will be revised at the time of the change, rather than wait for the annual review. To accomplish the review, the originator of the SOP or designated individual shall review a document and make any appropriate changes. An electronic copy of the document for review or change shall be sent by the QAA to the requestor or reviewer. Changes will be listed in the Revision/Change History section of the document. If no changes are made, the date of the annual review will be documented in the Revision/Change History section.

3.4.2. Forms

The R8L uses a variety of forms to document recurring activities. The process for form creation and approval is given in the *Controlled Document Review, Approval, Change, and Obsolete Procedure* (QSP-003). The process is similar to that for SOPs. Approved forms are available in read-only versions on the Laboratory's G: drive at G:\Lab QA\ Released Documents\.

3.5. Review of Analysis Requests

The process used to plan and document requests for services provided by the R8L are contained in SOP (GENLP-811), *Laboratory Services Request Procedure*. These services include, but are not limited to chemical analysis, biological testing, field support, technical assistance and supplies. The procedure is performed using the R8L Laboratory Information Management System (LIMS), the Region 8 Lotus Notes email system, and the Region 8 Avaya telephone system. Communication may also occur in person.

To summarize, an initial request for laboratory services is made by an EPA Project Manager. The initial contact is usually made through the yearly call for work spreadsheet that the laboratory circulates to previous customers and Managerial staff. Additionally, many project requests are received via phone calls between existing lab customers and laboratory project managers. Sometimes the initial request is made through an electronic initial contact form housed on the Region 8 intranet. The project is discussed at the R8L technical meetings. A Laboratory Project

Manager (LPM) is assigned, usually an analyst whose specialty is primarily requested. The scope of work is evaluated and analysts consult their calendars to verify the project can be accepted. If the work can be accomplished by the R8L, the LPM assigns the project an Laboratory Services Request (LSR) number and generates an LSR form using the Element LIMS system. The LPM is responsible for communicating with the customer and finalizing details such as specific methods, reporting limits, sampling dates, due dates, timesheet account codes, etc.

3.6. Subcontracting of Environmental Tests

The R8L does not subcontract analyses.

3.7. Purchasing Services and Supplies

The quality of purchased materials (i.e., reagents, standards, instrumentation, and equipment) used in the laboratory directly affects the quality of the analytical results produced. General supplies that are not involved in and do not affect the testing or services performed by the laboratory can be purchased from any appropriate vendor. All supplies and services that can affect the quality of the environmental tests performed or the health and safety of personnel at the laboratory shall be selected and purchased from approved vendors that can meet requirements and specifications defined in the methods concerned. These materials are controlled to ensure that laboratory or method-specific requirements are met.

Quality requirements are often specified in analytical methods. Specific SOPs are consulted prior to the performance of any analytical procedure to determine these requirements. The following is a summary of general procurement requirements imposed to ensure the generation of high quality data:

- The laboratory purchases reagents and solvents with purity requirements as established within the appropriate SOPs. General purpose reagents (i.e., acids, bases, sodium sulfate, etc.) are Analytical Reagent (AR) Grade, meaning that they conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS);
- Ultrapure reagents may be required in the method SOPs for some trace analyses;
- Upon receipt, the labels of chemicals, reagents, solvents and standards will be verified for correctness with the packing slip. In addition, verification that appropriate Material Safety Data Sheets (MSDS) and certificates of analysis / quality are received for all chemicals, reagents, and standards will be performed;
- Certificates of analysis / quality accompanying purchases shall be retained by the R8L for quality control tracking purposes for the lifetime of the purchase plus 1 year;
- All chemicals must be entered into the Vertere Chemical Inventory Management (ChIM) System upon receipt. Each chemical container, bottle, kit, or compressed gas must be clearly labeled with the ChIM bar code sticker.
- In the event an expiration date has not been assigned by the manufacturer, the expiration date will be assigned;
- Liquid, compressed, or bottled gases meet minimum specifications defined by the appropriate method SOP.

3.8. Service to the Client

The R8L will do everything possible to assure that its clients receive the best possible service, while maintaining confidentiality when required.

This may include, but is not limited to:

- Cooperating with clients in the clarification of analysis requests and the performance of said analyses;
- Providing clients access to the Laboratory to observe testing when requested;
- Providing any additional documentation concerning sample analyses that may be requested and / or required by the client;

- Advising, guiding and communicating with the client in technical matters, opinions and interpretations in regard to analyses performed or to be performed;
- Communicating with the client regarding any excursions in analyses being performed;
- Communicating with the client with regard to any delays that may result in the customer not receiving their analytical results in a timely manner;
- Notifying the client of any finding that casts doubt on the validity of results supplied to them.

3.9. Customer Feedback, Complaints, and Data Quality Inquiries

As part of its quality system, the R8L has established procedures to document non-conformances, complaints, corrective actions and maintain records of how quality issues, environmental compliance, and external complaints are resolved. These are outlined in the *Nonconformances, Complaints, Corrective and Preventative Actions Processing Procedure* (QSP-006). This procedure is applicable to non-conformances found by internal and external audits, quality assessments, environmental compliance issues, and external complaints or inquiries.

It is the responsibility of the laboratory analyst or technician identifying the nonconformity or receiving the complaint to begin the non-conformance process and to forward as much information as possible about the issue to the LQAO. This individual should also initiate a non-conforming item report. The QAA will record and track all Nonconforming Item / Complaint reports. The LQAO, or designee, will perform any initial investigation of the cause, including root cause of the issue and proposed corrective actions. The LQAO will review all Nonconforming Item / Complaint reports and verify that all appropriate corrective and / or preventative actions have been taken. The LQAO and Laboratory Director will review all investigations for appropriateness and completeness. An annual review of complaints, data quality inquiries, and nonconformances will be presented as part of the management review process.

3.10. Control of Nonconforming Environmental Testing

Procedures, method acceptance criteria and QC criteria are detailed in each test method/SOP. The analyst performing the test is responsible for evaluating test method/SOP compliance with respect to these parameters. Nonconforming issues should be evaluated and corrected prior to committing samples to analysis in a noncompliant measurement system. The analyst is responsible for identifying noncompliance issues and their impact on data quality in the case narrative.

3.10.1. **Identification of nonconformance** is typically made by the analyst; however, nonconformance can also be identified during the review process before release of the data, or during an internal or external audit.

3.10.2. **Once nonconformance is identified**, corrective actions should be taken immediately, together with any decision about the acceptability of the nonconforming work. If nonconformance is severe, the associated work should not continue without the authorization of the LQAO or Laboratory Director.

3.10.3. **Whenever departures from documented policies and procedures or from standard specifications result in a compromised analytical result**, the data is qualified. The reason for qualification is discussed in the case narrative.

3.10.4. **Samples occasionally fail to conform** to typical matrix expectations to such an extent that they are unable to be analyzed by the selected method without modification. If the analytical staff decides that an excursion from an SOP or test method is required, the procedure outlined below will be followed.

- The staff of the R8L will obtain approval from the client (i.e., EPA Project Manager) to perform the excursion, document any excursion from an SOP or test method, and technically justify the excursion.
- The analyst will seek advance approval from the EPA Project Manager to use her/his best professional judgment to deviate from established procedures as

appropriate to address specific analytical challenges. This occurs as part of the LSR process prior to sample receipt by the R8L. If the client gives advance approval for excursions, the analysts may make excursions from the analytical SOPs and test methods based on their best professional judgment without subsequently consulting the client. The client will be informed of these excursions in the case narrative that accompanies the data.

3.10.5. The client will be notified if any identified nonconformance with the test method/SOP significantly affects data quality.

3.11. Corrective Action

Corrective actions are those taken to eliminate the causes of an identified nonconformity, defect, or other undesirable situation. Most of these arise from isolated instances of QC outliers, errors due to calculation or transcription, or cases of abnormal instrument performance. At the first level, the individual analyst is responsible for performing all required QC checks, determining if the results for the QC items are within the acceptance criteria, and performing the appropriate corrective actions as specified in the relevant SOP or test method. For items identified during the peer review process, again it is the individual analyst who is responsible for performing the necessary corrective actions. In all cases, the QC failure must be identified in the case narrative and its impact on the data quality described for the data user. The affected sample data must be appropriately qualified in the data package, the narrative, and in LIMS.

If QA reviews, internal audits, or management reviews find outliers or errors of any particular type (QC sample failure, transcription error, calculation error, etc.) occurring at an abnormal frequency, the process in *Nonconformances, Complaints, Corrective and Preventative Actions Processing Procedure* (QSP-006) will be followed, including a corrective action report. Any R8L staff member may initiate a corrective action for issues that can affect data quality.

Corrective action may be initiated due to a variety of situations (e.g., failed proficiency testing (PT) results, client complaints, system audits, special outside request, equipment problems, quality problems with supplies). The corrective action process is given in the *Nonconformances, Complaints, Corrective and Preventative Actions Processing Procedure* (QSP-006).

3.12. Preventative Action

Preventive action is a proactive process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints. It aims to minimize or eliminate poor data quality or other nonconformances through scheduled maintenance and review.

Preventive actions includes, but are not limited to, review of QC data to identify quality trends, inclusion of quality topics at regularly scheduled staff and technical meetings, annual managerial reviews, regular instrument maintenance, and other actions taken to prevent problems.

Each analyst is responsible for ensuring that analytical instrumentation is in good working order prior to beginning analysis. Each analyst is also expected to consider and communicate instrument service considerations when scheduling sample load with the other analysts.

3.13. Control of Records

The R8L maintains a record system which produces unequivocal, accurate records that document all laboratory activities and complies with applicable regulations. Records may be in hard copy or electronic media. The record keeping system allows for historical reconstruction of all Laboratory activities that produced the analytical data. The laboratory retains on record all original observations, calculations and derived data, calibration records, and a copy of the final report. The hard copy of the project files are considered the official copy and are maintained on-site for a minimum of two years, after which time they are transferred to the National Archives - Federal Records Center where they are retained according to the records retention schedules EPA Records Schedule 018, Sampling and Analytical Data Files - Superfund Site-Specific and EPA Records Schedule 223, Sampling and Analytical Data Files. The overall records management system is governed by the EPA Records Management Policy, CIO 2155.1, June 8,

2009. In the event of laboratory closure, all records are transferred to the National Archives where they are maintained according to the records retention schedules.

3.13.1. Hard Copy Project Files: Each analytical project is identified by its LSR number and has both a hard copy project file and an electronic file. Where possible, all information placed in the project file will be original. In some instances, such as bound log books, it may be necessary to make copies; however, it is essential that the copy placed in the file be the exact copy of the original. The following hard copy information is maintained for all projects:

- A project folder containing the Chain of Custody (COC) sheets, the LSR, any field sample data, and other information such as shipping/receiving, the Final Report, Sample Disposal Letter, and QA Checklist/Record of Transmittal; and
- All raw data used in the decision making process for obtaining reported results including, but not limited to: QA/QC information, peer review form and checklists for all analyses, chromatograms, raw quantitation reports, calculations, any special notes concerning the project, copies of log book pages.

3.13.2. Electronic Project Files: The R8L uses the EPA internet and the R8L intranet to maintain its records.

- All sample information and analytical results are maintained on the R8L LIMS. This includes scanned copies of the COCs. The LIMS is used to create LSRs, final reports, and electronic data deliverables (EDDs) for each project.
- Project folders are maintained on the R8L intranet. At a minimum, these contain a copy of the LSR, scanned copies of the COCs, the final report in pdf format, the sample disposal letter, and the EDD, and other sample information relevant to the project.
- The Region 8 Lotus Notes email system is used to deliver the final report in pdf format, the sample disposal letter, and the EDD to the client. In addition, a project folder is created on the R8L share drive. It contains the final report in pdf format, the sample disposal letter, the EDD, and copies of email correspondence regarding the project in pdf format.
- Complete backups of these files occur daily.

3.13.3. QA/QC Records: The following QA/QC data shall be maintained by each LQAO or designee:

- Demonstrations of Capability (DOCs), Method Detection Limits (MDLs), and other similar data;
- PT results;
- internal and external audit records and responses;
- certification of thermometers and weights; and,
- corrective action log and corrective action records (i.e., completed forms, memos, etc.).

3.14. Internal Audits

The Region 8 LQAO shall conduct annual internal audits of R8L activities to verify that its operations continue to comply with the requirements of the quality system. Other R8L personnel may be tasked to assist in conducting the audits. The audits will generally verify that approved procedures are in place and being followed and that corrective actions are being performed by R8L personnel in a timely and responsive manner.

- The audits will include review of R8L documentation and procedures in each of the functional areas.
- The internal audits of each analytical area will include review of QA/QC files, PT results,

SOPs, log books, as well as sample preparation and analysis procedures.

- The internal audits of the sample custodial function will include review of SOPs, as well as sample log-in, sample storage and disposal procedures and documentation.
- Audit reports summarizing findings will be written and submitted to the Laboratory Director.
- If determined necessary as a result of an internal audit, corrective action(s) will be carried out as described in Section 3.10.

3.15. Management Reviews

At least once per year, Region 8 Laboratory Management Team shall conduct a review of the quality system to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and Laboratory operations. The review takes account of reports from Laboratory personnel, the outcome of recent internal audits, assessments by external bodies, the results of proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions and other relevant factors. The Laboratory Management Team shall consist of the Laboratory Director, the Health and Safety / Environmental Compliance Officer, the LQAO, and senior analysts from each of the functional areas.

- The management review will be summarized in a written report.
- If determined necessary as a result of the management review, corrective action(s) will be carried out as described in section 3.10.

4. TECHNICAL REQUIREMENTS

4.1. General

The R8L is an environmental testing laboratory. Testing is performed in accordance with specific test methods/SOPs. The Laboratory takes into account many factors to assure that testing is being performed in accordance with the test method/SOP requirements and that the results of the testing are correct and reliable. These factors include contributions from:

- personnel;
- accommodations and environmental conditions;
- environmental test methods and method validation;
- equipment;
- measurement traceability;
- sampling; and,
- handling of samples.

4.2. Personnel

The R8L has personnel with the necessary education, training, technical knowledge, and expertise to perform their job functions. Each Laboratory position has a job description identifying position title, relevant qualifications, minimum job requirements for the position, responsibilities, and reporting relationship. Personnel records are maintained by the Laboratory Director and by the Region 8 Human Resources Department.

4.2.1. Training

All R8L personnel are trained to perform their assigned tasks and made aware that the Laboratory follows the national EPA quality system and NELAC quality system requirements for environmental testing. This training is on-going and must be kept up-to-date. The training procedure is given in In-House Personnel Training Procedure (QSP-005). Note: This procedure does not pertain to national EPA or regional EPA training requirements or records.

Three levels of training are conducted:

- Quality System and Ethics Training - This training is performed by the LQAO or designated individual and describes the R8L quality system, including key quality system procedures with special emphasis on the quality manual. The importance of ethics and data integrity is included in this training. This training shall occur as follows: New employees within 30 calendar days of hire, and annually for all employees.
- Job-related Training – This training is given to employees before they perform job functions that, if done incorrectly, could adversely affect the product, test or service.
- Procedures, Methods, and Forms Training – This training shall be read, understood, and documented when required.

There may be some training required for all R8L personnel on an annual basis such as various health and safety related training courses; environmental management systems training which is not covered by this procedure.

All personnel are responsible for learning and complying with all QA/QC requirements that pertain to their organizational and technical functions. Training is documented on the *Training Report* form (QSF-001). It is the responsibility of the Quality Assurance Assistant to assure that the training records are updated and available in the training files.

4.2.2. Initial Demonstration of Capability (IDC)

All personnel must demonstrate capability in the activities and methods which they perform. Each analyst must demonstrate the ability to generate acceptable results by performing an IDC prior to unsupervised analysis of real world samples.

The IDC shall be performed on clean quality system matrix samples (ones with no target analytes or interferences present at concentrations that impact the results of the test method) and includes a demonstration of the analyst's ability to achieve a low background and the precision and accuracy required by the test method.

- To accomplish the IDC, the analyst must perform an MDL determination and prepare and analyze four replicates of a blank matrix spiked at mid-range.
- All method blank results must be less than the required method reporting limits or established laboratory reporting limits.
- The mean recoveries and the relative standard deviations for each parameter of interest in the four replicates are compared to specific method requirements or, in the absence of method requirements, established laboratory control limits for matrix spike / matrix spike duplicate samples to determine acceptance.
- When one or more of the tested parameters fails to meet the acceptance criteria, the analyst must locate and correct the source of the problem, repeat the analyses, and successfully meet all acceptance criteria before analyzing actual samples.
- For analytes which do not lend themselves to spiking, the IDC may be performed using quality control samples.
- An IDC must be completed each time there is a change in instrument type, personnel or method.
- Copies of the IDC results, data, etc. shall be identified, reviewed and approved by the laboratory director and the LQAO, and archived in the Quality Assurance files.
- A *Certification Statement* (QSF-002) shall be used to document the completion of each initial demonstration of capability, with a copy retained in the personnel training records for each analyst.

4.2.3. On-going Demonstration of Capability (DOC)

On-going Demonstration of Capability (DOC) shall be performed at least once per year for each analyst by any of the following:

- Acceptable performance of a blind sample.
- An initial test method evaluation or demonstration of capability.
- At least four consecutive laboratory control samples with acceptable levels of precision and accuracy.
- Analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.

The on-going Demonstrations of Capability shall be documented on the *On-Going Demonstration of Capability Record* form (QSF-003), with a copy retained in the personnel training records for each analyst.

4.3. Accommodations and Environment

The R8L is located in the Coors Technological Center, at 16194 West 45th Drive, Golden, CO 80403. The facility consists of six sections, A to F, with each section housing specific areas. Laboratory and office spaces are shared by the EPA and EPA's Environmental Services Assistance Team (ESAT) contractor. Access to the laboratory is controlled by electronically locked doors. Laboratory personnel have access badges and can enter the laboratory at doors on each wing. All other personnel must enter through the main entrance, with access electronically controlled.

The A Section consists of the laboratory administration, the computer LIMS, a library, conference rooms, a break room, and the ESAT offices. The A section of the laboratory is separated from the analytical part of the laboratory by electronically locked double doors. To visit outside of the A section, visitors must sign in and obtain a pass from the front desk and be escorted to the location requested.

The B Section is the main wing after passing through the double doors. This wing houses the four metals preparation and analysis laboratories including (B104 [ESAT], B106 [shared prep area], B108, and B110), plus the three wet chemistry laboratories (B111, B113, and B116). The chemical receiving laboratory (B102) is found in this section. The B Section houses one gas chromatograph/mass spectrometry (GC/MS) laboratory for volatiles analysis (B145) and one gas chromatographic (GC) laboratory (B138) for volatiles and semivolatiles analysis. Other operations in this section include asbestos analysis (B129 [ESAT]), transmission electron microscope (B147 [ESAT]), and the air (B124) and radiation (B125 and B127) chemistry laboratories.

The C Section is a separate wing located off the B section. Here are housed two microbiology (C102, C103), a macrobiology (C104), and three biology portions of the laboratory consisting of two toxicology laboratories (C116 and C117) and a general biology/PCR laboratory (C115). In addition, there is analyst office space.

The D Section is also a separate wing branching off the B Section containing the three organic preparation laboratories (D126, D127, and D128), two GC/MS semivolatile analysis laboratories (D104 and D124), a high performance liquid chromatograph (HPLC) and liquid chromatograph/ mass spectrometer (LC/MS) laboratory (D102), and a miscellaneous laboratory (D121). Also included is analyst office space.

The E Section is the third branch off the B Section. It contains the sample receiving area (E115), shipping and receiving rooms, a shop, general storage, the field equipment staging area, and the glassware cleaning/autoclave area (E105). Sample storage is accommodated in Room E115 by two large locked walk-in coolers and one locked cooler used for volatile storage only, and by several smaller refrigerators in individual laboratories for storage of standards and perishable supplies. The Region 8 Mobile Laboratory is stored in E-112.

The F Section is a separate building that houses the corrosive/reactive and flammable chemicals used in the laboratory and where the hazardous waste collected from the laboratory is stored for disposal (F103–F106).

The G Section is at the end of the B Section. It contains the general plant operations and acid neutralization areas.

The laboratory has ten E-pure systems in individual laboratories for delivery of 16 to 18 megohm-cm resistivity water. The de-ionized feed water for these systems is based in Room B118. All laboratories have appropriate safety equipment including hoods, safety shower and eyewash stations, fire extinguishers, first aid kits, personal protective equipment, and spill cleanup supplies.

4.4. Environmental Test Methods and Method Validation

Staff at R8L shall use appropriate methods and procedures for all environmental tests within the Laboratory's testing scope. The R8L staff has instructions on the use and operation of all relevant equipment and on the handling and preparation of samples where the absence of such instructions could jeopardize the results of environmental tests. All instructions, standards, manuals and reference data relevant to the work of the Laboratory are kept up-to-date and are readily available to personnel. Deviation from environmental test methods or procedures shall occur only if the deviation has been documented, is technically justified, and authorized.

4.4.1. SOPs

The R8L develops and maintains SOPs that accurately reflect all phases of current Laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods. SOPs are developed following the process outlined in Section 3.3.1.

4.4.2. Laboratory Test Methods/Technical SOPs

The R8L maintains a test method/SOP for each analysis type. In cases where modifications to the published method have been made (for example, where the referenced test method is ambiguous or provides insufficient detail), the Laboratory's procedures/acceptance criteria are clearly described. If there are program specific requirements, these, too, are identified. Each test method/SOP shall include the following:

- Title;
- Revision/Change history;
- Purpose;
- Scope and application;
- Summary of the method;
- Personnel qualifications and responsibilities
- Definitions;
- Interferences;
- Health, safety and cautions;
- Equipment and supplies;
- Reagents and standards;
- Quality control and quality assurance;
- Sample handling
- Sample preparation and analysis
- Instrument or method calibration
- Analysis
- Data acquisition, calculations and data reduction
- Data review and acceptance
- Computer hardware and software
- Troubleshooting
- Data and records management;
- Waste management and pollution prevention;
- References; and
- Tables, diagrams, flowcharts and QC data;

4.4.3. Selection of Methods

The R8L uses methods for environmental testing which meet the needs of the client and which are appropriate for the objectives of the project. Established methods published in international, national or regional documents shall preferably be used. The R8Lwill use the most current valid edition of a method unless it is not appropriate or possible to do so. Approved Quality Assurance Project Plan (QAPP) requirements supersede all other stipulations regarding chosen method. These should be identified and agreed upon during the LSR approval process (See Section 3.4). Prior to acceptance and institution of any method, a satisfactory IDC is required (See Section 4.2.2).

4.4.4. Laboratory-Developed Methods

The introduction of environmental test methods developed by the Laboratory for its own use shall be a planned activity and shall be assigned to qualified personnel equipped with adequate resources. Plans shall be updated as development proceeds and effective communication amongst all personnel involved shall be ensured. The method developed shall have been validated appropriately before use.

4.4.5. Non-Standard Methods

When it is necessary to use methods not covered by standard methods, these shall be subject to agreement with the client and shall include a clear specification of the client's requirements and the purpose of the environmental test. The method developed shall have been validated appropriately before use.

4.4.6. Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled. The R8L shall validate non-standard methods, laboratory-designed / developed methods, standard methods used outside their intended scope, and modifications of standard methods to confirm that the methods are fit for the intended use. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application. The Laboratory shall record the results obtained and the procedure used for the validation.

The range and accuracy of the values obtainable from validated methods (e.g., the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), as assessed for the intended use, shall be relevant to the client's needs.

4.4.7. Estimation of Uncertainty of Measurement

The Environmental Protection Agency, as well as the R8L, recognizes that the largest contributor to measurement uncertainty for environmental samples is related to the difficulty in obtaining a representative sample. Sampling plans are usually included in client QAPPs and are outside the control of the Laboratory.

Unless there are specific client requirements, the R8L shall use the nested hierarchical approach in estimating uncertainty of measurements. This approach is based on EURACHEM/CITAC Guide CG 4, "Quantifying Uncertainty in Analytical Measurement", and uses backward induction to "back-out" component uncertainties from quality control sample data. Using quality control data, the sources of variation that affect measurement uncertainty can be broken down into specific components. At its most basic application, the in-house, statistically-derived LCS control limits are used as an estimate of the minimum laboratory contribution to measurement uncertainty at a 99% confidence level.

In those cases where a well-recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the R8L will follow the test method and reporting instructions.

4.4.8. Control of Data

4.4.8.1. Corrections

Corrections to records shall be made using a single line out with the date and the signature or initials of the individual making the corrections. No changes shall be made with any technique that obliterates the original such as erasures or correction fluid. All records and corrections shall be in ink. Pencil shall not be used on analytical records.

4.4.8.2. Manual Peak Integration

Electronic data reduction is used for most of the analysis systems. Analysts are required to review the electronic data processing for accuracy and consistency with appropriate data reduction techniques. This includes peak integration. Some electronic reduction can result in incorrect actions by the system and for these instances, manual override and correction of the electronic data processing is appropriate. Examples of this may include such items as integration of an incorrect peak, errors in calculations, or misplacement of the baseline in peak integration. When manual override of the electronic process is deemed appropriate, corrections shall be done in accordance with sound analytical procedures, and analysts shall document the action taken and reason. The analyst and peer reviewer shall be in agreement regarding the integration decision. If they cannot agree, they will involve another experienced analyst, the LQAO, or the Laboratory Director to achieve a mutually acceptable decision.

When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations (i.e., the chromatogram obtained after the manual integration must be retained to permit verification of the rationale and results). This requirement applies to all analytical runs including calibration standards and QC samples. The person performing the manual integration must document the rationale for performing manual integration and identify that manual integrations were performed in the case narrative.

4.4.8.3. Data Review

Data generated by the R8L are checked for completeness, accuracy, precision, and quality conformance before being reported to the client. Corrective action may be taken at any stage of the data review process. This internal data verification process consists of data generation, reduction, and three levels of review. The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. Evaluation of data quality is based on the results of the QC data and the professional judgment of those conducting the review. This infusion of technical knowledge and experience is essential in ensuring that the data is of consistent high quality. Following are the steps taken by the Region 8 laboratory to ensure quality data in all of the deliverables:

4.4.8.3.1. The analyst is responsible for the **initial review**. This includes verifying the following items:

- Sample preparation and analysis information is correct and complete;
- Calibration data are scientifically sound, appropriate to the method, and completely documented;
- The appropriate SOPs were followed, and the LSR requirements were met;
- Blanks, controls, duplicates, spikes, and other QC data that may have been requested are within the limits specified by the SOP, the LSR, or the analytical method;
- Qualitative identification of sample components is correct;
- Quantitative results (calculations) are correct and complete;
- Documentation is complete for all anomalies in preparation and analysis, such as any special preparations, corrective actions, holding times; and
- The narrative has been completed, and results have been entered into LIMS; and they have been checked for errors and then signed.

4.4.8.3.2. The **second phase** of verification is completed by a second analyst or peer reviewer. A peer is someone who is familiar with the analytical procedure. A minimum of 10 percent of the data are reviewed. Priority is given to results that are above the RL. Checks of computer calculations and analyst calculations are performed on a random basis. Any questions or changes that may be required are directed to the original analyst. The reviewer and the analyst must agree on the results. This piece of the review is designed to ensure the following:

- All appropriate documentation has been followed (SOPs, LSRs, etc.);
- Calibration data are correct for the method used and completely documented;
- QC results are within the acceptable limits based on method, SOP, or LSR information;
- Qualitative identification of sample components is correct;
- Quantitative results (calculations) are correct and complete;
- Documentation is complete for all anomalies in preparation and analysis, any special preparations, corrective actions, holding times, and the like, and the run and extraction logs are reviewed for completeness; and
- Certificates of analysis and the analyst's narrative are checked for completeness and accuracy and also for typographical and grammatical errors.

4.4.8.3.3. Once the **final data package is complete**, it is transmitted to the QA Section for a final review and preparation of the final report. These reviews are to determine the following:

- Verify completeness and general accuracy of the data package;
- Check for typographical errors and inconsistencies in the report;
- Verify that all sample numbers, stations, descriptions, dates, and times match the chain of custody; and
- All analytical work has been completed.
- All errors are documented on the QA review sheet and corrected by the original analyst.
- Once this is complete, the LQAO or designee generates the final report and EDD using the LIMS. The final report in pdf format, the EDD, and the sample disposal letter are then transmitted to the client.

4.4.8.4. Computers, Automated Equipment, Microprocessors

The R8L uses the Element Data System laboratory information management system (LIMS) which is manufactured by Promium. The LIMS has an in-house system manager who is responsible for LIMS maintenance and trouble shooting. Only personnel with a valid need are allowed access to the LIMS by control of the computers on which the system is installed and through the use of password systems within the software. User capabilities are controlled to prevent unauthorized actions, and the LIMS maintains an audit trail of user actions.

The R8L uses the Newport Vertere Inventory System which is manufactured by AT/SCAN Ltd. The Vertere system is utilized for the maintenance of chemical and equipment inventories. The Vertere system has an in-house system manager who is responsible for Vertere system maintenance and trouble shooting. User capabilities are controlled to prevent unauthorized actions, and Vertere maintains an audit trail of major user actions.

Personal computer based data systems are used to operate most instruments. These systems often calculate results based on raw data from the analysis. The results calculated by the system are verified by manually checking the calculated results using the raw data reports. Hardware and software are updated as required to maintain operations. Only systems from reputable manufacturers that meet minimum acquisition specifications are purchased and used.

4.5. Equipment

The R8L is furnished with all items of sampling, measurement and test equipment required to correctly perform all environmental testing within the Laboratory's scope. Laboratory equipment is only used by authorized personnel. Equipment and the accompanying software used for testing are capable of achieving the accuracy required and comply with specifications relevant to the environmental tests concerned. All equipment is calibrated or checked before being placed into use to ensure that it meets Laboratory specifications and the relevant standard and/or manufacturer's specifications. If equipment must be relocated within the Laboratory, its performance will be similarly verified prior to placing the item back in service.

4.5.1. Equipment Inventory

The R8L has an inventory of state-of-the-art analytical equipment. The current inventory is maintained by the Laboratory Property Manager. The inventory records include:

- the identity of the item of equipment and its software;
- the manufacturer's name, type identification, and serial number or other unique identification;
- date received and date placed in service (if available);
- current location (where appropriate); and,
- condition when received (e.g. new, used, reconditioned).

A database of the inventory is maintained in Vertere. An additional listing of major analytical equipment and support equipment is maintained in LIMS.

4.5.2. Maintenance/Service

Proper maintenance of laboratory instrumentation is the key to both the longevity of the useful life of the instrument and its ability to provide reliable analyses. This need is provided by analytical staff for routine preventative maintenance with support services provided either by in-house maintenance or by vendor technicians, as appropriate. All staff members have the responsibility for insuring that primary maintenance is carried out on instrumentation. The primary elements of the equipment maintenance program are included below.

- All major equipment receives a daily use check, e.g., cooling fan operation, pump operation, indicator readings, mechanical checks, clean air filters, etc.;
- Routine preventive maintenance is performed on all major equipment;
- Records are kept in maintenance logs for all repairs;
- Instrument utilization records are maintained in the form of analysis logs;
- A conservative supply of critical spare parts is maintained for high-use instrumentation; and
- Vendor produced operation and maintenance manuals (where available) are maintained for all laboratory instrumentation.

4.5.3. Support Equipment

Support equipment includes secondary devices necessary to support Laboratory operations. All support equipment shall be maintained in proper working order, with records kept of all repair and maintenance activities including service calls. Balances, ovens, refrigerators, freezers, and water baths shall be checked for acceptable performance prior to use on each working day. The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used. All support equipment shall be calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use.

4.5.3.1. Incubators and Water Baths

If automatic temperature recorders are not used for an incubator, verified thermometers immersed in a bottle of liquid will be placed on the top and bottom shelves. An analyst will record the temperatures at least twice each working day (more frequently if required by the method/SOP) when the incubator is in use. If for any reason a unit is not being used to contain materials for which a specified temperature is required by method, policy, or procedure daily checks will not be made while it is not in use. Before a unit is placed back into use, a current temperature measurement will be taken for verification that it is at the proper temperature for use. This measurement must be documented in the temperature recording log, indicating that the unit has been placed back into active service and daily temperature checks must then resume. For water baths used in microbiology, temperature will be monitored and recorded at least once each working day while the unit is in use or as may be specified by the method. Temperature variations will be checked when incubators or water baths, used in microbiology, are loaded to capacity. This check will be documented. Water baths will be drained and cleaned periodically as recommended by the manufacturer, method or accepted practice.

4.5.3.2. Refrigerators/Freezers/Drying Ovens

Each working day that a refrigerator, freezer or drying oven is "in use", the temperature will be checked and documented either through automatic temperature monitoring or manually. If for any reason a unit is not being used to contain materials for which a specified temperature is required by method, policy, or procedure, daily checks will not be made while not in use. Before a unit is placed back into use, a current temperature measurement will be taken for verification that it is at the proper temperature for use. The temperature will be documented in the temperature recording log, indicating that the unit has been placed back into active service and daily temperature checks will resume Periodic check of refrigerator, freezer and drying oven contents will be conducted and outdated materials will be properly disposed.

4.5.3.3. Autoclaves

Each time an autoclave is in use and/or as required by the analytical method, the temperature will be checked and documented. At a minimum, contents, date, sterilization cycle and time, and maximum temperature will be recorded and a sterilization strip will be included with each use. Commercially produced *Stereothermophilis* strips will be used monthly to confirm the effectiveness of the autoclave cycle. A quarterly check of autoclave timing and an annual maintenance check to include a pressure check and calibration of the temperature will be performed.

4.5.3.4. Balances

The performance of each balance is verified daily or prior to use with reference weights previously checked against NIST traceable primary weights. The verification is documented in the appropriate analysis or balance log. Balances are cleaned and leveled as needed. Balance calibration and reference weight verification is performed annually by a certified vendor, and maintenance records are stored by the Laboratory by the QA Section.

4.5.3.5. Thermometers

Unless otherwise specified by the regulatory methodology, it is the policy of the R8L to use non-mercury thermometers wherever possible. Thermometers and other temperature measuring devices are checked against NIST traceable, calibrated thermometers annually and corrections are applied to each as needed.

4.5.3.6. Mechanical Dispensing Devices

Mechanical volumetric dispensing devices (except Class A glassware) shall be checked for accuracy at least every three months (quarterly). Pipet calibration is performed annually by a certified vendor, and maintenance records are stored by the Laboratory by the QA Section. Note: Glass microliter syringes are exempt from this requirement; however, such syringes used for volumetric dispensation must be tested for accuracy as instructed by the manufacturer.

4.5.4. NIST Traceable References

4.5.4.1. The QA Section maintains a set of reference thermometers and a reference weight set. These are used to verify the performance of other thermometers and weights in the R8L as needed.

4.5.4.2. The reference thermometers and reference weights are kept in padded cases which must be used when transporting these to work areas within the R8L.

4.5.4.3. Records of NIST traceability for thermometers and weights shall be filed with QA records in the QA Section.

4.5.5. Instrument Calibration

Calibration procedures vary depending on the instrument type and analysis. In general, it consists of an initial calibration followed by calibration verification and/or continuing calibration verification. The test methods/SOPs for each analysis performed in the Laboratory describe the calibration procedures, their frequency, acceptance criteria and the conditions that will require re-calibration.

4.5.6. Initial Calibration

An initial calibration curve with concentrations bracketing the analyte range of interest will be prepared at the beginning of the analytical process and at a frequency required by the specific test method/SOP.

Sample results must be quantified from the initial instrument calibration and may not be quantified from any continuing calibration verification unless otherwise allowed by regulation, test method, or program.

All initial calibrations must be verified with a standard obtained from a second manufacturer.

- When only one manufacturer of a standard exists, the use of a standard from a second lot from the same manufacturer is acceptable.
- Some analyzes require standards prepared from neat materials. If commercially prepared standards are not available, a second, independently prepared, in-house standard will be used for verification.

Traceability shall be to a national standard, when available.

Criteria for the acceptance of an initial calibration must be established and used, e.g., correlation coefficient or relative percent difference. The criteria used must be appropriate to the calibration technique employed.

Sample results not bracketed by initial instrument calibration standards (i.e., with concentrations within the calibration range or linear range) must be reported as having less certainty. This will be identified in the case narrative and the appropriate data qualifiers applied. The lowest calibration standard must be above the method detection limit. Noted exceptions to this rule include the following:

- Instrument technology (such as ICP/OE or ICP/MS) with validation techniques from manufacturers or test methods employing standardization with a zero point and a single point calibration standard; and
- Standardized validated methods which expressly permit quantitation to a designated percentage above the calibration curve high standard.

In these cases, the reporting limit (RL) will be confirmed by a low-level standard prepared at the RL. The R8L does not report values less than the RL. The individual test methods/SOPs specify, in detail, the calibration procedures used.

If the initial instrument calibration results are outside established acceptance criteria, corrective action must be taken. In all cases, the initial calibration must be acceptable, as specified in the test method/SOP, prior to analyzing samples.

4.5.7. Continuing Calibration Verification

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration is verified by continuing calibration verification. The details of the continuing instrument calibration procedure, calculations and associated statistics are included or referenced in each individual test method/SOP.

Continuing calibration verification must be performed at the beginning and end of each analytical batch. An exception to this policy is for internal standard methods (e.g., GC/MS). For these analyses, the continuing calibration verification is only required at the beginning of the analytical sequence. Additional calibration verifications may be performed. If required, the concentrations of the continuing calibration verification standards shall be varied within the established calibration range.

If continuing calibration verification fails, and routine corrective action procedures fail to produce a second consecutive calibration check within acceptance criteria, a new initial calibration curve should be constructed. Sample data associated with unacceptable calibration verification must be identified in the case narrative and reported as qualified data.

4.6. Measurement Traceability

The overall program of calibration and/or verification and validation of equipment shall be designed and operated so as to ensure that measurements made by the R8L are traceable to national standards of measurement. Calibration certificates shall indicate the traceability to national standards of measurement and shall provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The Laboratory shall maintain records of all such certifications.

Where traceability to national standards of measurement is not applicable, the Laboratory shall provide satisfactory evidence of correlation of results, for example by participation in a suitable program of inter-laboratory comparisons, proficiency testing, or independent analysis.

4.7. Sampling

Sampling is performed by the R8L staff and by samplers from other organizations. When performed by the R8L, samples are collected using standard field sampling techniques consistent with the parameter(s) being determined. The sampling techniques minimize the possibility of sample adulteration by either the sample collector or sampling device, and only trained individuals perform the sampling. The field sampling procedure is given in Field Sampling Procedures (FLDM-720). All sample containers are certified, contaminant-free containers or are triple-rinsed prior to use. No sample containers are reused. The field sampling includes field blanks and field duplicate samples. Recommended sample preservation and holding times are included in the Laboratory's SOPs.

When necessary, the R8L prepares and ships coolers and containers to support field sampling activities. All sample containers are certified, contaminant-free containers. Included are an adequate number of containers to allow for QC samples such as field duplicates and matrix spike / matrix spike duplicate samples. Trip blanks are included as appropriate. Cooler shipment is in accordance with applicable regulations and carrier requirements.

4.8. Handling of Samples

With the exception of emergency response samples which are conditionally exempt, all work is scheduled by the R8L prior to sample receipt. Samples from unknown sources or unknown projects should not be received.

Sample receiving begins with project planning. Administrative procedure *Planning and Scheduling Laboratory Services* (ADMNP-003) describes the process, dates, responsibilities, and assumptions used to plan, request, and schedule chemical, biological, field, and consultative services from the R8L. The *Laboratory Services Request Procedure* (GENLP-811) defines the process used to plan and document requests for projects accepted by the laboratory. The *Sample Receipt and Control Procedure* (GENLP-808) describes the processes associated with the receipt, login, storage, and disposal of environmental samples submitted to the R8L. All samples received by the R8L for chemical and/or microbiological analyses are controlled by these procedures. The following is a general summary. See the individual procedures for details.

4.8.1. Sample Receipt

Complete documentation of the sample collection and handling process is an extremely important aspect of a regulatory monitoring effort. Formal chain-of custody (COC) procedures provide for a written record of sample traceability, accountability, and serve to validate sample integrity.

Samples should not arrive at the R8L without an accepted LSR which identifies the project name, the projected number and types of samples, the types of analyses, and any other relevant information. Samples should be accompanied by COCs and/or Field Sample Data. At the time of receipt, the individual receiving the samples will sign and date the COCs and any other shipping documents and ensure that the samples are moved to Sample Receiving and Storage Room (Room E115).

The Sample Receiving and Storage Room is shared by EPA and ESAT personnel. There are two walk-in coolers for general samples (Coolers A and B) and separate coolers for volatile organic analysis (VOA) samples (Cooler C and D). Cooler A is used by ESAT and not covered by this document. Cooler B is used for general EPA sample storage. Coolers C and D are for volatile organic samples, with Cooler D reserved for drinking water samples. In addition, there is a Bio-Freezer in Room C103 for samples which have to be maintained at subzero temperatures. The items shared by EPA and ESAT personnel in Room E115 include the general facilities and access to the LIMS.

At the time of receipt, the individual performing sample login will follow this general procedure:

- Check the coolers and seals for integrity and any signs of damage;
- Open the coolers. Check the contents for preservatives (ice) and damage and check the sample temperature. The temperature, general sample condition and any discrepancies will be noted on the COC which accompanies the cooler;
- Remove the samples. Arrange in order and verify the label information on the sample containers with the COC. Any discrepancies should be noted on the COC;
- Enter the sample information into LIMS. Print the sample labels and verify that the Laboratory ID and bottle ID on the label matches the field ID on the bottle;

- Place the samples into trays labeled with the project name, LSR number, date received, and analyses. Place the trays into the appropriate coolers. Note: The trays provide segregation of the samples and secondary containment for them;
- Place the COCs and LSR into the project folder; and
- Notify the appropriate analysts that the samples have been received and are available for analysis.

Where there is any doubt as to a sample's suitability for testing, the client is consulted for further instructions before proceeding with sample analyses.

4.8.2. Sample tracking

Sample information and analytical requests are logged into the LIMS. Each sample is uniquely identified.

Samples are removed from the Sample Receiving Room by the individuals performing the analysis. This is documented in the *Sample Checkout Log* form (GENLF-007). Required information includes the analyst's name, project number, type of analysis, sample matrix, and date and time out. Subsequently, the date and time in is documented.

Following the procedures outlined in the individual test methods/SOPs, analysts may further identify sample results as matrix spikes (MS), matrix spike duplicates (MSD) and/or duplicates.

4.8.3. Sample Storage

Samples which require thermal preservation are stored under refrigeration at ± 2 °C of the specified preservation temperature. For samples with a specified storage temperature of 4 °C, storage at a temperature above the freezing point of water to 6 °C is considered acceptable for chemistry samples. Samples are stored in locked coolers in a manner that prevents cross contamination.

4.8.4. Sample Disposal

All samples, digestates, leachates and extracts or other sample preparation products are disposed of in accordance with Federal and State laws and regulations. If the sample supports litigation, disposal of the physical sample occurs only with the concurrence of the affected legal authority, sample data user and/or submitter of the sample.

All samples analyzed at the R8L may be disposed of only after all analyses have been completed unless otherwise specified by the Project Manager. A minimum of 60 days after the final results are reported is allowed before sample disposal.

The EPA Region 8 Laboratory Chemical Hygiene Plan (CHP) sets forth the waste management and disposal procedures.

4.9. Assuring the Quality of Environmental Test Results

The Laboratory has quality control procedures for monitoring the validity of all environmental tests and calibrations undertaken.

4.9.1. Proficiency Testing

The R8L participates in independent Proficiency Testing (PT) Studies as required for accreditation or more often as deemed necessary. Performance on these studies further indicates the effectiveness of the Laboratory's day-to-day quality control procedures.

4.9.2. Standard Reference Materials and Certified Reference Materials

Standard Reference Materials (SRMs) and Certified Reference Materials (CRMs) are utilized to determine method/analytical performance as deemed appropriate.

4.9.3. Significant Figures / Rounding Rules

All reports are generated using the LIMS, and the number of significant figures and the rounding rules are built into the reports. In general, only three figures are reported. This corresponds to those that are known definitely, with one additional figure that may have some degree of uncertainty, which is true for many chemical measurements.

Rounding-off is performed when reporting the analytical results, but should not be done when recording the experimental data. Rounding should be performed only after arriving at the final result in the calculation. Rounding off numbers involves dropping digits that are not significant. If the insignificant digits are 5 or greater, the preceding number will be increased by one; if the digits to be dropped are less than 5, the preceding number will not be altered.

Rounding is never appropriate to meet QC criteria. For example, the requirement is ≤ 25%, 25.1% fails. Similarly, if the requirement is ≥ 5%, 4.95% fails.

4.9.4. Essential Quality Control Procedures

The R8L has quality control procedures for monitoring the validity of the environmental tests it undertakes. The data acquired from quality control procedures are used to estimate the quality of the analytical data, to determine the need for corrective action in response to identified deficiencies, and to interpret results after corrective action procedures are implemented. Each test method/SOP includes a quality control section which addresses the minimum QC requirements for the procedure. The essential QC procedures may differ for each individual test method but in general are described below.

4.9.4.1. Chemical Testing

Negative Control - Method Performance

Method blanks are prepared and analyzed at a minimum of one per batch of 20 or fewer samples per matrix type per sample extraction or preparation test method. The results of these samples are used to determine batch acceptance.

Positive Control - Method Performance

Laboratory control samples (LCSs) are prepared and analyzed at a minimum of one per batch of 20 or fewer samples per matrix type per sample extraction or preparation method. An exception to this rule involves analytes for which spiking solutions are not available, such as total solids, temperature and dissolved oxygen. The results for method performance samples are used to determine batch acceptance. Also know as laboratory fortified blank (LFB) or blank spike (BS).

Sample Specific Controls

The Laboratory determines the effect of the sample matrix on method performance. This procedure involves the analysis of matrix specific QC samples and is designed as a data quality indicator for a specific sample using the designated test method. Matrix spikes alone cannot be used to judge Laboratory performance.

- Surrogate compounds are added to all samples, standards, and blanks for all organic chromatography test methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery generally indicates a problem with the sample matrix and is reported to assist in data assessment.
- Matrix spikes (MSs)/matrix spike duplicates (MSDs) are prepared and analyzed at a minimum of one per batch of 20 or fewer samples per matrix type per sample extraction or preparation method. An exception to this rule involves analytes for which spiking solutions are not available, such as pH, temperature, dissolved oxygen, and turbidity. Additionally, sometimes the sample volume supplied is inadequate to perform

MS/MSD analyses. Poor performance with a matrix spike generally indicates a problem with the sample composition, and not the Laboratory analysis, and is reported to assist in data assessment.

- In general, all reportable components are included in the spike mixes. However, in cases where the components interfere with accurate assessment, the test method has an extremely long list of components or components are incompatible, a representative number of the listed components may be used. The selected components of each spiking mix represent all chemistries, elution patterns and masses and include permit-specified analytes and other client requested components.
- Laboratory duplicates may be used to evaluate analytical precision and are prepared and analyzed as required by method or SOP. Poor performance in the duplicates generally indicates a problem with the sample composition and is reported to assist in data assessment. In general, it is better to measure something rather than nothing when evaluating analytical precision, and measurement uncertainty increases as one gets closer to the detection limit. For these reasons, the Laboratory preference is to use MS/MSD pairs to evaluate precision rather than duplicate pairs for organic analyses. This will be superseded by method specific requirements.

Detection and Reporting Limits

A Method Detection Limit (MDL) study shall be performed initially (prior to the analysis of any samples) as part of the method validation. The Laboratory MDLs will be updated annually or with a significant change in instrument type, matrix, or test method as per 40 CFR Part 136, Appendix B, where applicable. MDLs determined by any other procedure will be as documented in the method and/or SOP.

Specific guidance on the appropriate frequency of MDL studies may be specified in the method or required by a program.

An MDL study is not performed for any component for which spiking solutions are not available, such as total suspended solids, total dissolved solids, total solids, pH, color, temperature, dissolved oxygen and physical tests. For these types of analytes, the detection limit shall be determined as specified in the method and/or SOP.

MDL study results shall be maintained by the QA Section.

Quantitation limits shall be established as per the test method and/or SOP. In all cases these shall be greater than the corresponding method detection limits and establish the Laboratory reporting levels. All reporting levels are confirmed with a reporting level check standard. Results are not reported below the reporting level

Data Reduction

Data shall be reduced according to method specifications, using all applicable procedures and equations as provided in each individual method. The procedures used for data reduction, such as the use of linear regression, shall be documented in the individual test method/SOP.

Quality of Standards and Reagents

The quality of chemicals, reagents, solvents, standards, and gases used in the Laboratory is determined by the sensitivity and specificity of the analytical techniques being used. Chemicals, reagents, solvents, standards, and gases of lesser purity than specified by a test method/SOP will not be used.

Selectivity

Absolute and relative retention times aid in the identification of components in chromatographic analyses and help evaluate the effectiveness of a column to separate constituents. Acceptance criteria for retention time windows are documented in each method SOP. A confirmation is performed to verify compound identification when positive results are detected on a sample from a location that has not been previously tested. Such confirmations are performed for organic analytes except when the analysis involves use of a mass spectrometer. Acceptance criteria for mass spectral tuning are contained in the test method/SOP.

Constant and Consistent Test Conditions

The Laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used. Glassware shall be cleaned to meet the sensitivity requirements of the test method. Any cleaning and storage procedures that are not specified by the test method shall be documented in Laboratory records and SOPs.

4.9.4.2. Microbiological Testing

Sterility Checks and Blanks

The R8L shall demonstrate that the sample containers, media and reagents used in microbiological testing have not been contaminated through improper handling or preparation, inadequate sterilization, or environmental exposure.

Positive Controls

Positive culture controls demonstrate that the medium can support the growth of the target organism(s), and that the medium produces the specified or expected reaction to the target organism(s).

Negative Controls

Negative culture controls demonstrate that the medium does not support the growth of non-target organisms or does not produce the typical positive reaction of the target organism(s).

Test Variability/Reproducibility

For test methods/SOPs, including chromofluorogenic and fluorogenic tests, that specify quantification of colony-forming units using empirically-derived most probable number tables, analyst counts are conducted once each month on at least one positive sample. When two analysts are available, each analyst shall count positive wells on the same multi-well tray or plate. Counts must be within 10% difference to be acceptable. When only one microbiologist is available, the same multi-well tray or plate shall be counted twice by the analyst, with no more than 5% difference between the counts.

Test Performance

All growth and recovery media must be checked to assure that the target organism(s) respond in an acceptable and predictable manner. To ensure that analysis results are accurate target organism identity shall be verified as specified in the method, e.g., by use of the completed test, or by use of secondary verification tests.

Data Reduction

The calculations, data reduction and statistical interpretations specified by each test method/SOP shall be followed.

Quality of Standards, Reagents and Media

The Laboratory shall ensure that the quality of the reagents and media used is appropriate for the test concerned.

Selectivity

In order to ensure identity and traceability, reference cultures used for positive and negative controls shall be obtained from a recognized national collection, organization, or manufacturer recognized by the NELAP Accrediting Authority. Microorganisms may be single use preparations or cultures maintained by documented procedures that demonstrate the continued purity and viability of the organism.

Constant and Consistent Test Conditions

Laboratory facilities and support equipment shall meet the appropriate quality specifications in the test method/SOP.

4.10. Reporting the Results

The results of environmental tests carried out by the R8L shall be reported accurately, clearly, unambiguously and objectively, and in accordance with any specific instructions in the test method/SOP. The environmental tests are performed primarily in support of EPA Region 8 programs such as RCRA, Superfund, Drinking Water, UST/LUST, CERCLA, and other initiatives.

4.10.1. Test Reports

Results are reported to the client electronically, unless requested otherwise. Electronic test results reported to the client include the following:

- Data release memo from the analysts, LQAO, and Laboratory Director (or their Designees) authorizing release of the data from the Laboratory;
- Case narrative prepared by the analysts summarizing the samples received, test methods, QC notes with identification of noncompliance issues and their impact on data quality, and an explaining any data qualifiers applied to the data;
- Report by parameter for each sample;
- Electronic data deliverable (EDD) of the sample results; and,
- Sample disposal letter.

For those tests for which the R8L will be NELAC accredited, all requirements of the current NELAC Standard will be met. This will be documented and acknowledged for the client in the case narrative. If a NELAC accredited test has not met the requirements of the current NELAC Standard, reasons and/or justification meeting the approval of the LQAO will be provided in the case narrative.

4.10.2. Preliminary Test Results

Sometimes in order to meet client needs, preliminary data is reported before data and report review are complete. When preliminary reporting of data is necessary, each part of the process listed below will be observed.

- All preliminary reports will be emailed or faxed so that there is a paper trail for what was reported.
- For each major project, there will be an email/communication on file saying that the client wants to receive preliminary reports and understands the possibility that reports may change following more thorough review.
- Preliminary data reports become part of the hard copy historical package once a data pack is completed. (This does not mean it will be part of the final report going to the client.) It is the responsibility of the person issuing the preliminary results to include a copy of the faxed or emailed results transmission.
- There is a higher risk of error pursuant to distributing preliminary results which have not undergone review by a second analyst. Therefore, the following statement will be added to every electronic preliminary results report.

- "The information in this report is being supplied to you at your request as 'Preliminary Results'. Results have not undergone the same level of review as a final report. Once all reviews have taken place, it is possible that results in the final report may vary from those in this report."

4.10.3. Project Files

Each individual project file contains the completed data package for each parameter tested. The project files consist of the following:

- Transmittal memo(s), QA review checklist, report by parameter for each sample; sample disposal letter, all relevant forms and checklists including: COCs and Field Sample Data sheets, and LSR;
- All raw data used in the decision making process for obtaining reported results including, but not limited to: QA/QC information, chromatograms, raw quantitation reports, calculations, any special notes concerning the project, copies of log book pages; correspondence (memos, e-mail or other documents relevant to the project).

4.10.4. Amendments to Test Reports

Material amendments to a test report after issue shall be made only in the form of a further document, or data transfer. All amended reports shall be uniquely identified with the amendment number at the top and bottom of each page and the reason for the amendment shall be identified in the case narrative.

5. REFERENCES

5.1. Ingersoll, W.S., Environmental Analytical Measurement Uncertainty Estimation – Nested Hierarchical Approach, Defense Technical Information Center # ADA396946, 2001.National Environmental Laboratory Accreditation Conference, *2003 NELAC Standard*, June 2003.

5.2. *Region 8 Quality Management Plan*, U.S. EPA Region 8, Denver, CO, December 2008.

5.3. Title 40: Code of Federal Regulations. *Protection of Environment*, Office of the Federal Register, National Archives and Records Administration. Washington, D. C.

5.4. U.S. EPA, *Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality-Related Documents,* EPA QA/G-6, April 2007.

5.5. U.S. EPA, *Manual for the Certification of Laboratories Analyzing Drinking Water*, Fifth Edition, EPA 815-R-05-004, January 2005.

5.6. NELAC Standards, Chapter 5, June 5, 2003. EPA/600/R-04/003.

6. TERMS and DEFINITIONS

AB: Accrediting Body

ANSI: American National Standards Institute

ASQC: American Society for Quality Control

ASTM: American Society for Testing and Materials

BLK: Blank – an aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other sample analysis. Blanks are used to detect contamination during sample handling preparation and/or analysis. Blanks types include bottle, equipment, field, instrument, preparation / method / calibration / laboratory reagent, storage, and trip.

BS: Blank spike or laboratory fortified blank – an aliquot of reagent water to which known quantities of the test method analytes are added in the laboratory. It is analyzed exactly as a sample.

°C: degrees Celsius

CAL: Calibration standard – a solution prepared from the primary standard solution. The calibration standards are used to calibrate the instrument.

CAS: Chemical Abstract Service

COC: Chain of custody

CCV: Continuing calibration verification – a calibration standard, generally of midpoint concentration for all elements, which is analyzed to verify that the system remains in calibration.

CCB: Continuing calibration blank – a calibration blank analyzed to verify that the system remains in calibration.

CRA: Reporting limit check standard – a low-level standard prepared at the reporting limit.

CWA: Clean Water Act

DI: Deionized water

DOC: Demonstration of Capability

DUP: Laboratory duplicates – two sample aliquots taken from the same sample in the analytical laboratory and analyzed separately with identical procedures to determine analytical precision.

EPA: Environmental Protection Agency

ESAT: Environmental Services Assistance Team

g/L: grams per liter

GC/MS: gas chromatography/mass spectrometry

ICP: Inductively coupled plasma

ICP-MS: inductively coupled plasma-mass spectrometry

ICV: Initial calibration verification – a solution prepared from a primary standard that is from a different source than that used for the initial calibration. It is used to verify the validity of the initial calibration. Also know as a quality control sample (QCS).

ICB: Initial calibration blank – a calibration blank analyzed as part of the calibration sequence.

IS: Internal standard

ISO/IEC: International Organization for Standardization/International Electrochemical Commission

LCS: Laboratory control sample – A sample matrix, free from the analytes of interest, spiked in the laboratory with known amounts of analytes or a material containing known and verified amounts of analytes and analyzed exactly as a sample. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of the measurement system. Also know as laboratory fortified blank (LFB)or blank spike (BS).

LDR: Linear dynamic range – the limit of quantitative results. At the upper limit of the LDR, analytical results are within ± 10% of the true value.

LIMS: Laboratory information management system – a computer database where sample results and all relevant data are stored.

LQAO: Laboratory quality assurance officer

LSR: Laboratory services request

LOD: Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific.

LOQ: Limit of Quantitation – The minimum level, concentration, or quantity of a target analyte that can be reported with a specified degree of confidence.

MDL: Method detection limit

MQL: Method quantitation limit – The lowest concentration standard that is analyzed establishes the MQL.

MRL: Minimum reporting level – A value below which the laboratory does not report results. The MRL is always greater than the MDL. For this laboratory, the MRL is set at the MQL.

mg/Kg: milligrams per kilogram

mg/L: milligrams per liter

MS: Matrix spike or laboratory fortified sample matrix – an aliquot of an environmental sample to which known quantities of the target analytes are added in the laboratory. It is analyzed exactly as a sample. Also known as sample spike or laboratory fortified sample matrix (LFSM).

MSD: Matrix spike duplicate or laboratory fortified sample matrix duplicate – a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. The duplicate sample is prepared simultaneously with the matrix spike sample, and each is spiked with identical, known concentrations of target analytes. Also know as sample spike duplicate or laboratory fortified sample matrix duplicate (LFSMD).

NELAC: National Environmental Laboratory Accreditation Conference

NELAP: National Environmental Laboratory Accreditation Program

NIST: National Institute of Standards and Technology

PT: Proficiency Test(ing)

Primary standard: a stock solution, usually obtained from a source outside of the laboratory, used to prepare secondary standards.

Secondary standard: a solution prepared in the laboratory from the primary standards and used for initial, continuing, and other standards as needed.

QAM: Quality assurance manual

QAPP: Quality assurance project plan

QA: Quality assurance

QC: Quality control

RCRA: Resource Conservation and Recovery Act

%R: Percent recovery The recovery of spiked analytes in clean (or sample) matrix. It is used to assess accuracy (or matrix effects).

RL: Reporting limit

RSD: Relative standard deviation

SARA: Superfund Authorization Recovery Act

SDWA: Safe Drinking Water Act

SD: Serial dilution – analysis of a sample which has been diluted to a concentration one-fifth of the original to confirm that there is no interference in the original sample analysis.

SOPs: Standard operating procedures

SRM: Standard reference material – A sample matrix containing test method analytes or a solution of method analytes that is used to fortify reagent water. It is obtained from a source different from the calibration source and is analyzed exactly as a sample. SRMs are used to check the performance of the measurement system.

SVOC: Semivolatile volatile organic compound

TNI: The NELAC institute

µg/L: micrograms per liter

VOC: Volatile organic compound

7. APPENDICES

7.1. Appendix A - R8L Analytical Capabilities.

Appendix A - R8L Analytical Capabilities‡

Analysis

DOC No.	Document/Method Name
Field Methods & Procedures (FLDM)	
FLDM-705	Field Sampling of Aquatic Macroinvertebrates Procedure
FLDM-720	Field Sampling Procedures
FLDM-721	Sediment Characterization Procedure
FLDM-722	Field Flow Measurements Procedure
FLDM-723	Algae Sampling Procedures
FLDM-730	Benthic Algae Field Sampling Procedure
Inorganic Chemistry Analysis Methods, Tests, Procedures, etc (INORM)	
INORM-001	Total Dissolved Solids by Conductivity Calculation
INORM-302*	**Determination of Total Alkalinity Using the DL50 Mettler Autotitrator**
INORM-303	Determination of Total Suspended Solids Gravimetric
INORM-307*	**Determination of Turbidity by Nephelometry**
INORM-310*	**Automated Determination of Fluoride, Chloride, Nitrate, Nitrate, Phosphorus, and Sulfate Using the Dionex Ion Chromatograph**
INORM-316	Automated Determination of Ammonia-Nitrogen Using the Lachat Flow Injection Analyzer
INORM-320	Digestion and Analysis to Total Phosphorus Using the All American Electric Pressure Sterilizer and Alpkem Segmented Flow Analyzer
INORM-323	Determination of Total Organic Carbon and Total Nitrogen Using the Shimadzu TOC-TN analyzer
Metals Analysis Methods, Tests, Procedures, etc. (METLM)	
METLM-201*	**Determination of Trace Elements by ICP-MS Using EPA Method 200.8**
METLM-203*	**Determination of Mercury Using Perkin Elmer FIMS Analyzer**
METLM-208	Determination of Mercury Using Nippon MA 200 Direct Hg Analyzer
METLM-210*	**Determination of Elements Using the Optima 5300 DV ICP-OES**
Microbiology Analysis Methods, Tests, Procedure, etc. (MICM)	
MICM-001	Air Density Testing for Heterotrophic Bacteria, Quality Control Procedure
MICM-002*	**SimPlate, Single- and Multi-dose, and Most Probable Number Methods for Quantification of Heterotrophic Bacterial in Aqueous Samples.**
MICM-003	Media Preparation
MICM-004	Microbiology Labware Cleaning and Sterilization
MICM-005	Pour Plate Preparation
MICM-006	Control Culture Preparation
MICM-007	Reagent Water Quality Control Tests
MICM-008	Heterotrophic Plate Count
MICM-009	DPD Method for Analysis of Free Total and Combined Chlorine in Reagent Water
MICM-010	Specific Conductance Determination of Water Samples Using the Orion Model 115 Conductivity Meter
MICM-602*	**Colilert Enzyme Substrate Method to Detect the Presence of Total Coliform Bacteria and Escherichia coli in Drinking Water**
MICM-624*	**Colilert Quanti-Tray 2000 Method for Quantification of Total Coliforms and E-Coli in Water**

Analysis

DOC No.	Document/Method Name
Organic Chemistry Analysis Methods, Tests, Procedure, etc. (ORGM)	
ORGM-001	Determination of Pharmaceuticals and Personal Care Products in Water by Direct Aqueous Injection LC/MS/MS
ORGM-002	Determination of 1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-chloropropane (DBCP) Using Method 8011 - SIM
ORGM-003	Determination Volatile Organic Compounds Using EPA Method 524.3
ORGM-004	Determination of Dissolved Methane, Ethane and Propane in Water by Equilibrium Headspace GC/FID Analysis
ORGM-005	Determination of Chlorophylls a and b in Freshwater Algae Using High Performance Liquid Chromatography with Visible Wavelength Detection and Fluorometry (EPA Method 447.0).
ORGM-006	Determination of Waste Indicator Compounds in Water by GC/MS
ORGM-007	Determination of BTEX, MTBE, 1,2-DCA, Naphthalene, and TPH/GRO Using 8260C
ORGM-008	Determination of Volatile Orgainics in soils using Headspace GC/MS
ORGM-500	Determination Volatile Organic Compounds Using EPA Method 524.2
ORGM-501*	**Determination of Volatile Organic Compounds Using EPA Method 8260B**
ORGM-502*	**Determination of Semivolatile Organic Compounds Using EPA Method 525.2**
ORGM-505*	**Determination of EDB, DBCP, and 1,2,3-TCP Using Microextraction**
ORGM-506*	**Determination of BTEX, MTBE, Naphthalene, and TPH/GRO Using 8021B and 8015B Modified**
ORGM-507	Determination of 1,2-Dichloroethane Using 8021B
ORGM-508*	**Determination of Diesel Range Organics Using 8015B Modified**
ORGM-509	Determination of Polychlorinated Biphenyls (PCB's) in Soil by Accelerated Solvent Extraction (EPA Method 3545) and Dual Capillary Column Gas Chromatography with micro-ECD (EPA Method 8082)
ORGM-512*	**Determination of Diquat and Paraquat in Drinking Waters by Solid Phase Extraction and HPLC with Ultraviolet Detection**
ORGM-513*	**Determination of Glyphosate in Drinking Water by Aqueous Direct Injection, Postcolumn Derivatization and Fluorescence Detection**
ORGM-514*	**Determination of N-Methylcarbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection HPLC with PCD and Fluorescence Detection**
ORGM-515*	**Determination of Semivolatile Organic Compounds Using Method 8270**
ORGM-520	Determination of Pesticides and Herbicides in Water by Liquid/Liquid Extraction and GC/MS-SIM
ORGM-550	Determination of Pesticides in Water by Direct Aqueous Injection LC/MS/MS
Sample Preparation Methods/Procedures (SAMPM)	
SAMPM-102*	**Open Container Digestion**

‡ Current as of April 2011

*** Procedures for NELAP accreditation**

Appendix II – Analytical Methods, Sample Containers, and Preservation

Appendix II-Analytical Methods, Sample Containers and Preservation

Sampling Locations	Analytical Method/ Parameter	# Samples (including field duplicates)	Sample Volume	Container Size and Type	Preservation	Max. Holding Time
All Sample Locations See Table 1	8260 Frac 2013 in Water, EPA Method 8620B	**24** Fixed Locations plus 1 Field Replicate, 1 MS, 1 MSD, 1 Trip Blank, and 1 Field Blank	29 x 3 vials x 40 ml per vial	Sterile 40 ml clear glass VOA vials	Ice to 2-6°C	14 Days
	8270 Frac Full in Water, EPA Method 8270D	**24** Fixed Locations plus 1 Field Replicate, 1 MS, 1 MSD, and 1 Field Blank	28 x 1000 mL	Sterile 1000 ml Amber Glass	Ice to 2-6°C HCl added at Lab	7 days
	Ammonia – R8 in Water, EPA Method 350.1 Total Phosphorus (TP)-R8 in Water, EPA Method 365.4	**24** Fixed Locations plus 1 Field Replicate and 1 Field Blank	26 x 125 mL	125mL plastic	Ice to 2-6°C H2SO4	28 days
	Anions by cap IC in Water, EPA Method 300.0	**24** Fixed Locations plus 1 Field Replicate and 1 Field Blank	26 x 125 mL	125 mL plastic	Ice to 2-6°C	28 days
	Gasoline-Low in Water, EPA Method 8021/8015	**24** Fixed Locations plus 1 Field Replicate, 1 MS, 1 MSD, 1 Trip Blank, and 1 Field Blank	29 x 3 vials x 40 ml per vial	Sterile 40 mL VOA vial glass amber	Ice to 2-6°C HCl	14 days
	Diesel-Low-1000ml in Water, EPA Method 8015	**24** Fixed Locations plus 1 Field Replicate, 1 MS, 1 MSD, and 1 Field Blank	28 x 1000 mL	Sterile 1000 mL glass amber	Ice to 2-6°C HCl	7 days

Appendix II-Analytical Methods, Sample Containers and Preservation

Sampling Locations	Analytical Method/ Parameter	# Samples (including field duplicates)	Sample Volume	Container Size and Type	Preservation	Max. Holding Time
All Sample Locations See Table 1	DOC – R8 in Water, EPA Method 415.2	**24** Fixed Locations plus 1 Field Replicate plus 1 Field Blank	26 x 125 mL	125 mL plastic	Ice to 2-6°C HCl / filtered	28 days
	TOC – R8 in Water, EPA Method 415.1 / SM 4	**24** Fixed Locations plus 2 Field Replicate plus 1 Field Blank	27 x 125 mL	125 mL plastic	Ice to 2-6°C HCl	28 days
	ICP Dissolved Metals – R8 in Water, EPA Method 200.7/6010 ICP-MS Dissolved Metals – R8 in Water, EPA Method 200.8/6020	**24** Fixed Locations plus 1 Field Replicate and 1 Field Blank	26 x 125 mL	125 mL plastic	Ice to 2-6°C HNO_3 / filtered	180 days
	ICP Total Metals – R8 in Water, EPA Method 200.7/6010 ICP-MS Total Metals – R8 in Water, EPA Method 200.8/6020	**24** Fixed Locations plus 1 Field Replicate, 1 Field Blank, and 1 Trip Blank	27 x 250 mL	250 mL plastic	Ice to 2-6°C HNO_3	180 days
	Nutrients-NNP in Water, EPA 365.3 (Orthophosphate as P) (Nitrite as N) (Nitrate as N)	**24** Fixed Locations plus 1 Field Replicate and 1 Field Blank	26 x 4L	1 gallon cube container and 1 quart cube container	HNO_3 at Lab	None
	Field Parameters per Equipment Manufacturer Instructions – Temperature pH Conductivity Dissolved Oxygen Stream Discharge (3 sites – FM-2, MC-2, and BH-1)	**24** Fixed Locations	In Situ	N/A	N/A	Analyze immediately

Appendix III – U.S. Geological Survey Surface-Water Quality Field Notes

U. S. GEOLOGICAL SURVEY SURFACE-WATER QUALITY FIELD NOTES

Station No. ____________

NWIS Record No. ____________

Station No. ____________ Station Name ____________ Field ID ____________

Sample Date ________ Mean Sample Time ______ Time Datum _____ (eg. EST, EDT, UTC) End Date ________ End Time ______

*Sample Medium: WS WSQ OAQ *Sample Type: 9 (regular) 7 (replicate) 2 (blank) 1 (spike) ________

*Sample Purpose (71999): 10 (routine) 15 (NAWQA) 20 (NASQAN) 25 (NMN) 30 (Benchmark) ____________

* see last page for additional codes

*Purpose of Site Visit (50280): 1001 (fixed-frequency SW) 1003 (extreme high flow SW) 1004 (extreme low flow SW) 1098 (NAWQA QC) _____

QC Samples Collected? Y N Blank Replicate Spike Other____________

Project No. ____________ Project Name____________

Sampling Team ____________ Team Lead Signature ____________ Date ________

START TIME ________ GAGE HT ______ TIME ________ GHT ______ TIME ________ GHT ______ TIME ________ GHT ______ END TIME ________ GHT ______

FIELD MEASUREMENTS

Property	Parm Code	Method Code http://water.usgs.gov/usgs/owq/Forms/Fieldmeasurement_parametersmethods.doc	Result	Units	Remark Code	Value Qualifier	Null Value Qualifier	NWIS Result-Level Comments
Gage Height	00065			ft				
Discharge, instantaneous	00061			cfs				
Temperature, Air	00020	THM04 (Thermistor) THM05 (Thermometer)		˚C				
Temperature, Water	00010	THM01 (Thermistor)		˚C				
Specific Conductance	00095	SC001 (Contacting Sensor)		µS/cm				
Dissolved Oxygen	00300	LUMIN (Luminescent) MEMBR (Amperometric) SPC10 (Spectrophotometric)		mg/L				
Barometric Pressure	00025	BAROM (Barometer)		mm Hg				
pH	00400	PROBE (Electrode)		units				
Alkalinity, filtrd, incr. Alkalinity, filtrd, Gran	39086 29802	TT061 (Digital Titrator) TT062 (Buret) TT056 (Digital Titrator) TT057 (Buret)		mg/L				
Carbonate, filtrd, incr. Carbonate, filtrd, Gran	00452 63788	ASM01(Digital Titrator) ASM02(Buret) ASM03(Digital Titrator) ASM04(Buret)		mg/L				
Bicarbonate, filtrd, incr. Bicarbonate, filtrd, Gran	00453 63786	ASM01(Digital Titrator) ASM02(Buret) ASM03(Digital Titrator) ASM04(Buret)		mg/L				
Hydroxide, filtrd, incr. Hydroxide, filtrd, Gran	71834 29800	ASM01(Digital Titrator) ASM02(Buret) ASM03(Digital Titrator) ASM04(Buret)		mg/L				
Turbidity [see attachment for codes and units]								

SAMPLING INFORMATION

Parameter	Pcode	Value	Information
Sampler Type	84164	*see last page for proper codes— consider type of sampler and material*	Sampler ID:
Sampling Method	82398	10 EWI; 20 EDI; 30 single vertical; 40 multiple vertical; other ____________	
Sampler bottle/bag material	84182	Plastic Bag (11) Teflon• Bag(12) Glass Bottle(20) Pastic Bottle (21) Teflon•Bottle (22) other (30)	
Sampler Nozzle material	72219	plastic (2) Teflon• (3) Brass (1)	
Sampler Nozzle Diameter	72220	3/16" (3) 1/4" (4) 5/16" (5)	
Sampler Transit Rate	50015	feet/second	
Velocity to Calculate Isokinetic transit rate	72196	feet/second	
Depth to Calculate Isokinetic transit rate	72195	feet	
Splitter Type	84171	*See last page for codes* ________	Splitter ID:
Hydrologic Condition	N/A	A Not Determined; 4 Stable, low stage; 5 Falling stage; 6 Stable, high stage; 7 Peak stage; 8 Rising stage; 9 Stable, normal stage	

BAG SAMPLER EFFICIENCY TEST

Test	Duration Sampler Collected Water (seconds)	Sample Volume Collected (milliliters)
1		
2		
3		
Mean	(72217)	(72218)
Bag Sampler Efficiency *(See last page)*		%

Observations [Codes: 0=none; 1=mild; 2=moderate; 3=serious; 4=extreme]

Oil-grease (01300) ___ Detergent suds (01305) ___ Floating garbage (01320) ___ Floating algae mats (01325) ___

Floating debris (01345) ___ Turbidity (01350) ___ Atm. Odor (01330) ___ Fish kill (01340) ___

Gas Bubbles (01310) ___ Sewage Solids (01335) ___ Floating Vegetation (84178) ___ Ice Cover(01355) ___

COMPILED BY: ____________ **CHECKED BY:** ____________ **LOGGED INTO NWIS BY:** ____________

Station No. ____________

SAMPLING CONDITIONS

Stream width (0004): ___________ ft mi **Left bank** ___________ Right bank __________ft Ice cover _____% Ave. ice thickness _____ in.

Number of Sampling points (00063): ____

Stations on cross-section (distance from LEFT RIGHT bank)____ ____ ____ ____ ____ ____ ____ ____ ____ ____ ____ ____ ____ ____

Sampling location: wading cableway boat bridge upstream downstream side of bridge _____ ft mi above below gage __________

Sampling site: pool riffle open channel braided backwater Bottom: bedrock rock cobble gravel sand silt concrete other __________

Stream color: brown green blue gray clear other ________ Stream mixing: well-mixed stratified poorly-mixed unknown other_______

Weather (00041) ______________

wind speed (00035) _____ mph *temperature-* very cold cool warm hot

No. days since last rainfall event (72053) _________

Observations:

Sample Comments (for NWIS; 300 characters max.):

LABORATORY INFORMATION *Sample Set ID* _____

SAMPLES COLLECTED (check all that apply):

Nutrients: ___WCA ___FCC ___FCCVT ___FCA Major cations: ___FA ___RA Major anions: ___FU

Trace elements: ___FA ___RA ___CU Mercury: ___FAM ___RAM ___Wis. Hg Lab Lab pH/SC/ANC: ___RU

Organics: ____GCC filtered __ unfiltered __ ___BGC ___C18 ___ Kansas OGRG Lab _____PEST ____PHARM ____HUN ____HFL

VOC: ______GCV (____ vials) Suspended solids: ___SUSO Turbidity: ___TBY Methylene Blue Active Substances: ___MBAS Color: ___RCB

Carbon: ___TPCN filter1-vol filtered_____mL filter2-vol filtered_____mL filter3-vol filtered_____mL ___DOC ___TOC

Stable isotopes: ___FUS ___RUS Radio-chemicals: ___FUR ___RUR ___SUR ___FAR ___RAR ___CUR

___BOD ___COD Chlorophyll: ____ CHL Algae: ____ Invertebrates: ___IQE ___IQL ___IQM ___IRE Fish tissue: ___TBI

Ultraviolet Absorbing Substances: ___UAS

Other:______________ (Lab____________) Other:______________ (Lab____________) Other:______________ (Lab____________)

Other:______________ (Lab____________) Other:______________ (Lab____________) Other:______________ (Lab____________)

Suspended sediment: ____ CONC. S/F SIZE [No. bottles _____]

Microbiology: ______________________________ (Lab________________) Date shipped: ___________

Date sediment sample shipped: __________ Sediment Lab: ____________________

Comments:

Date shipped:___________ Laboratory ____________________ Date shipped_____________ Laboratory_________________

Date shipped:___________ Laboratory ____________________ Date shipped_____________ Laboratory_________________

****Notify the NWQL in advance if shipping potentially hazardous samples—phone 1-866-ASK-NWQL or email LabLogin@usgs.gov**

Calibrated by:____________________ Location:____________________ Station No. ____________
Date:______________ Time:____________

METER CALIBRATIONS and FIELD MEASUREMENTS

TEMPERATURE Meter make/model ______________ S/N ____________ Thermistor S/N __________ Thermometer ID ________

Calibration criteria: ± 0.2 °C for thermistors

Local Meter

Lab Tested against NIST Thermometer/Thermistor? Y N Date: ____________ ±__________ °C

Measurement Location: SINGLE POINT AT ______ ft DEEP STREAMSIDE _____ FT FROM LEFT RIGHT BANK VERTICAL AVG/MEDIAN OF _____ PTS

Field Readings # 1 _____ #2 _____ #3 _____ #4 _____ #5 _____ **MEDIAN:**_______ °C Method Code _____ Remark ___ ___ Qualifier __

SPECIFIC CONDUCTANCE Meter MAKE/MODEL ______________ S/N ____________ Sensor ID ______________

Sample: CONE SPLITTER CHURN SPLITTER SINGLE POINT AT ______ ft DEEP VERTICAL AVG. OF _______ POINTS

LOCAL METER ID:______________ AUTO TEMP COMPENSATED METER? Y N CORRECTION FACTOR APPLIES? Y N CORRECTION FACTOR:______

Std Value µS/cm	Std Temp	SC Before Adj.	SC After Adj.	Vendor Lot No.	NWIS Parameter Code (see last page)	NWIS* Lot No.	Expiration Date

Calibration Criteria: ± 5 % for SC ≤100 µS/cm or 3% for SC >100 µS/cm ***NWIS Lot Numbers are available at: http://wwwnwql.cr.usgs.gov/qas.shtml?ConductivityStds_home**

Field readings #1_____ #2_____ #3_____ #4_____ #5 _____ **MEDIAN:** _______ **µS/cm** Method Code _______ Remark ___ ___ Qualifier ___

DISSOLVED OXYGEN Meter MAKE/MODEL ____________________ S/N ______________

Sensor Type: Amperometric Luminescent Spectrophotometer Sensor ID ______________Local Meter ID__________

Calibration Method: Air-Saturated Water Water-Saturated Air

Sample: SINGLE POINT AT _______ ft DEEP VERTICAL AVG. OF _____ POINTS BOD BOTTLE OTHER____________ Stirrer Used? Y N

Calibration Temperature °C	Barometric Pressure mm Hg	DO Table Reading mg/L	Salinity Correction Factor	DO Before Adjustment mg/L	DO After Adjustment mg/L

Zero DO Check _________mg/L Adj. to _________ mg/L Date: ____________

Thermister Check? Y N Date__________

Barometer Calibrated? N Y Date: ______________ Time: _______

Phase Degrees/Slope/Gain/Scale Factor (100%)______________(Zero)________

Calibration Criteria: ± 0.2 mg/L **DO saturation** _______ **%**

Field readings #1______ #2_____ #3_____ #4_____ #5 _____ **MEDIAN:** _______ **mg/L** Method Code _______ Remark ___ ___ Qualifier ____

---

pH Meter MAKE/MODEL ______________ S/N ____________ Electrode ID ____________ Type: GEL LIQUID OTHER ________

Sample: FILTERED UNFILTERED CONE CHURN SPLITTER SINGLE POINT AT _______ ft DEEP VERTICAL AVG. OF ____ POINTS

TEMPERATURE CORRECTION FACTORS APPLIED TO BUFFERS? Y N

pH BUFFER	BUFFER TEMP	THEORETICAL pH FROM TABLE	pH BEFORE ADJ.	pH AFTER ADJ.	SLOPE	MILLIVOLTS
pH 7						
pH ___						
CHECK pH ___						

pH Buffer	Vendor Lot No.	NWIS* Lot No.	Expiration Date
pH 7 (99173)			
pH 10 (99171)			
pH 4 (99172)			

Calibration Criteria: ± 0.1 pH units, ± 0.3 if SC <75us/cm ***NWIS Lot Numbers are available at: http://wwwnwql.cr.usgs.gov/qas.shtml?Buffers_home**
Millivolts: pH 7 –10 t0 +10 , pH4 +165 to +195 mV, pH 10 –165 to –195 mV
Slope Acceptance Criteria: 95% to 102%

Field Readings #1_________ #2_________ #3_________ #4_________ #5 _________ **MEDIAN:** _______ **Units** Remark ___ ___ Qualifier ___

Station No. ________________

TURBIDITY Meter make/model ____________________ **S/N** ________________ Type: turbidimeter submersible spectrophotometer

Sample: pump discharge line flow-thru chamber single point at _______ ft blw LSD MSL MP Sensor ID ________________

Sample: Collection Time: ________ Measurement Time: ________ Measurement: In-situ/On-site Vehicle Office lab NWQL Other __________

Sample diluted? Y N Vol. of dilution water ________ mL Sample volume ________ mL

	Lot Number or Date Prepared	Expiration Date	Concentration ________ (units)	Calibration Temperature °C	Initial instrument reading	Reading after adjustment
Stock Turbidity Standard						
Zero Standard *(DIW)*						
Standard 1						
Standard 2						
Standard 3						

TURBIDITY VALUE = $A \times (B+C) / C$

where:

A= TURBIDITY VALUE IN DILUTED SAMPLE
B= VOLUME OF DILUTION WATER, mL
C= SAMPLE VOLUME, mL

Calibration Criteria:

≤ 100 Turbidity units	± 0.5 turbidity units or ± 5% of the measured Value, whichever Is greater
> 100 Turbidity units	± 10%

Field Readings #1 __________ #2 __________ #3 __________ #4 __________ #5 __________

MEDIAN ________ **Parameter Code** _________ FNU NTU NTRU FNMU FNRU FAU FBU AU METHOD CODE __________ Remark ___ ___ Qualifier ___ ___

CROSS SECTION NOTES Barometric pressure = ________ mm Hg

Station	ft from left bank (00009) or ft from right bank (72103)	Time	Gage ht ft (00065)	Depth to Bottom at this station ft (81093)	Depth of measure-ment ft (00003)	Temp °C (00010) ____ THM01	SC µS/cm (00095) ____ SC001	DO mg/L (00300) ____ (Method Code)	DO sat %	pH units (00400) ____ PROBE	Turbidity ________ (________) ____ (Method Code)	Chl A Units (________)	NWIS Record No.
1													
2													
3													
4													
5													
6													
7													
8													
9													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													
21													
22													
23													
24													

NOTES:

ALKALINITY/ANC

BEGINNING H_2O TEMP. __________ °C
Specific Conductance____________µS/cm

PH	ΔPH	VOL ACID DC OR mL	ΔVOL ACID DC OR mL	ΔPH / ΔVOL ACID

End H_2O temp. __________ °C
Specific Conductance____________µS/cm

FIRST TITRATION

DATE ______________ INITIALS _______________
BEGIN TIME____________ END TIME____________
ALKALINITY/ANC ______________ mg/L* AS $CaCO_3$
BICARBONATE _________________mg/L* AS HCO_3^-
CARBONATE _________________mg/L* AS CO_3^{2-}
HYDROXIDE _________________mg/L* AS OH^-
ACID: 1.6N 0.16N 0.01639N OTHER:_______
ACID LOT NO. ________EXPIRATION DATE _________
CORRECTION FACTOR: 1.01 __________
ACID DELIVERY: DIGITAL COUNTER BURET
SAMPLE VOLUME: ________mL FILTERED UNFILTERED
METHOD: INFLECTION POINT GRAN
STIRRING METHOD: MAGNETIC MANUAL

BEGINNING H_2O TEMP. __________ °C
Specific Conductance____________µS/cm

PH	ΔPH	VOL ACID DC OR mL	ΔVOL ACID DC OR mL	ΔPH / ΔVOL ACID

End H_2O temp. __________ °C
Specific Conductance____________µS/cm

SECOND TITRATION

DATE ______________ INITIALS _______________
BEGIN TIME____________ END TIME____________
ALKALINITY/ANC ______________ mg/L* AS $CaCO_3$
BICARBONATE _________________mg/L* AS HCO_3^-
CARBONATE _________________mg/L* AS CO_3^{2-}
HYDROXIDE _________________mg/L* AS OH^-
ACID: 1.6N 0.16N 0.01639N OTHER:_______
ACID LOT NO. ________EXPIRATION DATE _________
CORRECTION FACTOR: 1.01 __________
ACID DELIVERY: DIGITAL COUNTER BURET
SAMPLE VOLUME: ________mL FILTERED UNFILTERED
METHOD: INFLECTION POINT GRAN
STIRRING METHOD: MAGNETIC MANUAL

Go to the Alkalinity Calculator for a complete listing of reporting rules, parameter codes, method codes, etc:
http://or.water.usgs.gov/alk

Alkalinity/ANC pH Meter Calibration

Meter make/model:	S/N	
Calibration Location:		
Electrode No.		
Electrode type GEL LIQUID		
	pH 7	pH ____
BUFFER TEMPERATURE		
THEORETICAL FROM TABLE		
pH BEFORE ADJUSTMENT		
pH AFTER ADJUSTMENT		
SLOPE		
MILLIVOLTS		
MANUFACTURER LOT NUMBER		
NWIS LOT NUMBER		

Field titration by:________________

Checked by:_______________

Station No. ____________

QUALITY-CONTROL INFORMATION

PRESERVATIVE, BLANK WATER and SPIKE NWIS LOT NUMBERS

NWIS lot numbers are available at: http://wwwnwql.cr.usgs.gov/qas.shtml?nfssqa_certificates

Description	Parameter Code	Expiration Date	Manufacturer Lot Number	NWIS Lot Number
4.5N H_2SO_4 (NUTRIENTS AND DOC)	99156			
7.5N-7.7N HNO_3 (METALS&CATIONS)	99159			
6N HCl (Mercury)	99158			
1:1 HCl (VOC)	99157			
18N H_2SO_4 (COD and Phenol)	99155			
Inorganic Blank Water	99201			
Organic Blank Water	99203			
VOC/Organic Blank Water	99205			
Spike	99104			

FILTER LOT NUMBERS

Filter descriptions with parameter codes require NWIS LOT NUMBERS available at http://wwwnwql.cr.usgs.gov/qas.shtml?filters_home

Filter Type	Pore Size (microns)	Manufacturer's Lot Number	Parameter Code	NWIS Lot Numer
Capsule	0.45		99206	
Disc	0.45		99206	
142 mm GFF (organics)	0.70			
Syringe (organics)	0.70		99207	
25 mm GFF (organic carbon)	0.70			
142 mm membrane (inorganics)	0.45			

QC SAMPLES

Sample Type	NWIS Record No.	Sample Type	NWIS Record No.	Sample Type	NWIS Record No.
Equip Blank ____	____________	Sequential ____	____________	Trip Blank ________	____________
Field Blank ____	____________	Spike ____	____________	Other ________	____________
Split ____	____________	Concurrent ____	____________	Other ________	____________

NWQL Schedules/lab codes (QC Samples) ________ ________ ________ ________ ________ ________

COMMENTS:____

(Circle appropriate selections)

99100 Blank-solution type
- 10 Inorganic grade (distilled/deionized)
- 40 Pesticide grade (OK for organics and organic carbon)
- 50 Volatile-organic grade (OK for VOCs, organics, and organic carbon)
- 200 Other

99101 Source of blank water
- 10 NWQL
- 40 NIST
- 55 Wisconsin Mercury Lab
- 140 EMD Chemicals
- 150 Ricca Chemical Company
- 200 Other

99105 Replicate-sample type
- 10 Concurrent
- 20 Sequential
- 30 Split
- 40 Split-Concurrent
- 50 Split-Sequential
- 200 Other

99102 Blank-sample type
- 1 Source Solution
- 30 Trip
- 40 Sampler
- 50 Splitter
- 80 Equipment (done in non-field environment)
- 90 Ambient
- 100 Field
- 200 Other

99111 QC sample associated with this environmental sample
- 1 No associated QA data
- 10 Blank
- 30 Replicate Sample
- 40 Spike sample
- 110 Cross-section information stored
- 100 More than one type of QA sample
- 200 Other

A complete list of fixed-value codes can be found online at:
http://wwwnwis.er.usgs.gov/currentdocs/index.html

99106 Spike-sample type
- 10 Field
- 20 Lab

99107 Spike-solution source
- 10 NWQL

99108 Spike-solution volume, mL ____________

99112 Purpose, Topical QC data
- 1 Routine QC (non-topical)
- 10 Topical for high bias (contamination)
- 20 Topical for low bias (recovery)
- 100 Topical for variability (field equip)
- 110 Topical for variability (field collection)
- 120 Topical for variability (field personnel)
- 130 Topical for variability (field processing)
- 140 Topical for variability (shipping&handling)
- 200 Topical for variability (lab)
- 900 Other topical QC purpose

REFERENCE LIST FOR CODES USED ON THIS FORM

The complete list of fixed-value codes can be found online at: http://wwwnwis.er.usgs.gov/currentdocs/index.html

Sample Medium Codes

WS	Surface water
WSQ	Quality-control sample (Replicate, Spike)
OAQ	Blank

Value Qualifiers

e	see field comment
f	sample field preparation problem
k	counts outside the acceptable range

Null-value Qualifiers

e	required equipment not functional or available
f	sample discarded; improper filter used
o	insufficient amount of sample
p	sample discarded; improper preservation
q	sample discarded; holding time exceeded
r	sample ruined in preparation

84164 Sampler Type

100	Van Dorn Sampler
110	Sewage Sampler
125	Kemmerer Bottle
3044	US DH-81
3045	US DH-81 With Teflon Cap And Nozzle
3047	Sampler, Frame-Type, Plastic Bottle W/Reynolds Oven Bag
3048	Sampler, Frame-Type, Teflon Bottle
3049	Sampler, Frame-Type, Plastic Bottle
3050	Sampler, Frame-Type, Plastic Bottle W/Teflon Collapsible Bag
3051	US DH-95 Teflon Bottle
3052	US DH-95 Plastic Bottle
3053	US D-95 Teflon Bottle
3054	US D-95 Plastic Bottle
3055	US D-96 Bag Sampler
3057	US D-99 Bag Sampler
3058	US DH-2 Bag Sampler
3060	Weighted-Bottle Sampler
3061	US WBH-96 Weighted-Bottle Sampler
3070	Grab Sample
3071	Open-Mouth Bottle
3080	VOC Hand Sampler
4010	Thief Sampler
4115	Sampler, point, automatic
8000	None
8010	Other

84171 Splitter type, field, code

10	Churn splitter, plastic, 8 liter, cooler-type spigot
20	Churn splitter, plastic, 14 liter, cooler-type spigot
30	Churn splitter, plastic, 8 liter, cubitainer-type spigot
40	Churn splitter, plastic, 14 liter, cubitainer-type spigot
50	Churn splitter, fluoropolymer, 8 liter (future development)
60	Churn splitter, fluoropolymer, 14 liter, US SS-1
70	Cone splitter, plastic
80	Cone splitter, fluoropolymer
90	Sieve, wet
100	Sieve, dry
110	Riffle splitter (Jones)
200	Other

Sample Type Code

9	Regular
7	Replicate
2	Blank
1	Spike
3	Reference
B	Other QA
H	Composite

71999 Sample Purpose

10	Routine
15	NAWQA
20	NASQAN
25	National Monitoring Network
30	Benchmark
40	SW Network
60	Lowflow Network
70	Highflow Network
110	Seepage Study
180	Cross-Section Variation

Bag Sampler Intake Efficiency (IE)

$$IE = K \times \frac{(V/T)}{Vs}$$

IE=Intake Efficiency
T=Mean Duration Sampler Collected Water (P72217)
V=Mean Sample Volume Collected (P72218)
Vs=Mean Stream Velocity (P72196)
K = 0.1841 for 3/16" nozzle
K = 0.1036 for 1/4" nozzle
K = 0.0663 for 5/16" nozzle

NWIS Lot Number Parameter Codes* for Conductance Standards

Parameter Code	Standard Value µS/cm, KCl
99160	50
99161	100
99162	250
99163	500
99164	750
99165	1000
99166	2500
99167	5000
99168	10,000
99169	25,000
99170	50,000

Time Datum Codes

Time Zone	Std Time Code	UTC Offset (hours)	Daylight Time Code	UTC Offset (hours)
Hawaii-Aleutian	HST	-10	HDT	-9
Alaska	AKST	-9	AKDT	-8
Pacific	PST	-8	PDT	-7
Mountain	MST	-7	MDT	-6
Central	CST	-6	CDT	-5
Eastern	EST	-5	EDT	-4
Atlantic	AST	-4	ADT	-3

82398 Sampling Method

10	Equal Width Increment (EWI)
15	Multiple Verticals, non-isokinetic, equal widths and transit rate
20	Equal Discharge Increment (EDI)
25	Timed Sampling Interval
30	Single Vertical
40	Multiple Verticals
50	Point Sample
55	Composite, multi-point samples
70	Grab Sample (Dip)
8030	Grab Sample At Water-Supply Tap

50280 Purpose of Site Visit

1001	Fixed frequency, surface-water
1002	Storm hydrograph, surface-water
1003	Extreme high flow, surface-water
1004	Extreme low flow, surface-water
1005	Diurnal, surface-water
1006	Synoptic, surface-water
1098	NAWQA surface-water quality control
1099	Other, surface-water
3001	Occurrence Survey, bed sediment or tissue
3002	Spatial Distribution Survey, bed sediment or tissue
3003	Synoptic Study, bed sediment or tissue
3098	Bed-sediment or tissue quality control
3099	Other, bed sediment or tissue

Dissolved Oxygen

AZIDE	Azide-modified Winkler
INDIGO	Spectrophotometer, indigo carmine
INDKT	Field Kit, indigo carmine, visual
LUMIN	Luminscence sensor
MEMB2	Amperometric, Membrane (DODEC)
MEMBR	Amperometric, Membrane electrode
RHODA	Field Kit, Rhodazine-D, visual
SPC10	Spectrophotometer, Rhodazine-D
WINKL	Winkler titration

Parameter and method codes for field measurements: http://water.usgs.gov/usgs/owq/Forms.html

***NWIS Lot numbers and Certificates of Analysis: http://wwwnwql.cr.usgs.gov/qas.shtml?nfssqa_certificates**

National Field Manual: http://water.usgs.gov/owq/FieldManual/

Alkalinity Calculator, Alkalinity/ANC parameter and method codes: http://or.water.usgs.gov/alk/reporting.html

Appendix IV – U.S. Geological Survey Groundwater Quality Field Notes

USGS science for a changing world

U. S. GEOLOGICAL SURVEY GROUNDWATER QUALITY FIELD NOTES

NWIS RECORD NO ______________

Station No. ______________ Station Name ______________ Field ID ______________

Sample Date ______________ Mean Sample Time (watch) ______________ Time Datum ________ (eg. EST, EDT, UTC)

Sample Medium ______ Sample Type _____ Sample Purpose (71999) _____ Purpose of Site Visit (50280) _____ QC Samples Collected? Y N

Project No. ______________ Project Name ______________

Sampling Team ______________ Team Lead Signature ______________ Date ________

FIELD MEASUREMENTS

Property	Parm Code	Method Code	Result	Units	Remark Code	Value Qualifier	Null Value Qualifier	NWIS Result-Level Comments
Water Level (see p. 8 for codes and units)								
Flow Rate	00059			gal/min				
Sampling Depth	00003			ft				
Depth to top of sampling interval	72015			ft blw lsd				
Depth to bottom of sampling interval	72016			ft blw lsd				
Temperature, Air	00020	THM04 (Thermistor) THM05 (Thermometer)		°C				
Temperature, Water	00010	THM01 (Thermistor) THM02 (Thermometer)		°C				
Specific Conductance	00095	SC001 (Contacting Sensor)		µS/cm				
Dissolved Oxygen	00300	SPC1 (Spectrophotometer) LUMIN (Luminescent MEMBR (Amperometric)		mg/L				
Barometric Pressure	00025	BAROM (Barometer)		mm Hg				
pH	00400	PROBE (Electrode)		units				
ANC, unfiltered, incr. ANC, unfiltered, Gran	00419 29813	TT065 (Digital counter) TT066 (Buret) TT058 (Digital counter) TT059 (Buret)		mg/L				
Alkalinity, filtrd., incr. Alkalinity, filtrd., Gran	39086 29802	TT061 (Digital counter) TT062 (Buret) TT058 (Digital counter) TT059 (Buret)		mg/L				
Carbonate, filtrd., incr Carbonate, filtrd., Gran	00452 63788	ASM01 (Digital counter) ASM02 (Buret) ASM03 (Digital counter) ASM04 (Buret)		mg/L				
Bicarbonate, filtrd., incr. Bicarbonate, filtrd., Gran	00453 63786	ASM01 (Digital counter) ASM02 (Buret) ASM03 (Digital counter) ASM04 (Buret)		mg/L				
Hydroxide, filtrd., incr. Hydroxide, filtrd., Gran	71834 29800	ASM01 (Digital counter) ASM02 (Buret) ASM03 (Digital counter) ASM04 (Buret)		mg/L				
Turbidity [see attachment for codes]								
Redox potential (Eh)	63002			mvolts				
Hydrogen sulfide odor detected?	71875	SNIF1 (sniff test, acidified sample) SNIF2 (sniff test, non-acidified sample)	#	Yes No	M detect U non-detect			Sample acidified beforehand? yes no
Hydrogen sulfide, unfiltered, measured	99119	ISE01 (electrode) KIT01 (Chemetrics) KIT02 (Hach)		mg/L				

SAMPLING INFORMATION

Parameter	Pcode	Value	Information
Sampling Condition*	72006		Sampler/Pump Type (make/model): ______________
Sampling Method*	82398		Pump/Sampler ID: ______________ Sampler Material: stainless steel pvc teflon other ______________
Sampler Type*	84164		Tubing Material: teflon plastic tygon copper other ______________
***see p. 8 for values**			Filter type(s): capsule disc 142mm 25mm GFF membrane

COMPILED BY: ______________ **CHECKED BY:** ______________ **LOGGED INTO NWIS BY:** ______________

Date ______________ **Date** ______________ **Date** ______________

FIELD ID ______________ Station No. ______________

Aquifer name ______________________________ Depth pump set at: __________ ft blw lsd msl mp

Sampling point description ______________________________

GW Color: *brown gray blue green yellow other* ______________________________

GW Clarity: *clear turbid muddy other* ______________________ *Foaming: Yes No*

*Sand Present: Yes No If yes, color of sand: Black Brown Tan Yellow Gray Other*__________

GW Odor: Yes No *describe* ______________________________

Sample in contact with: atmosphere oxygen nitrogen other _____

Weather: ***sky-*** clear partly cloudy cloudy ***precipitation-*** none light medium heavy snow sleet rain mist ________

wind- calm light breeze gusty windy est. wind speed ________ mph ***temperature-*** very cold cool warm hot

Observations:

Sample Comments (for NWIS; 300 characters max.):

LABORATORY INFORMATION *Sample Set ID* ____

SAMPLES COLLECTED:

Nutrients: ___WCA ___FCC ___FCA Major cations: ___FA ___RA Major anions: ___FU Trace elements: ___FA ___RA

Mercury: ___FAM ___RAM ___Wis. Hg Lab Lab pH/SC/ANC: ___RU

VOC: _____GCV (____ vials) Suspended solids: ___SUSO Turbidity: ___TBY Methylene Blue Active Substances: ___MBAS Color: ___RCB

Carbon: ___DOC ___TOC

Radon: ___RURCV (Radon sample collection time:_____) Stable isotopes: ___FUS ___RUS

Radiochemicals: ___FUR ___RUR ___SUR ___FAR ___RAR ___BOD ___COD

Other:____________ (Lab____________) Other:____________ (Lab____________) Other:____________ (Lab____________)

Other:____________ (Lab____________) Other:____________ (Lab____________) Other:____________ (Lab____________)

Microbiology: ______________________________ (Lab ____________)

Comments: ______________________________

Date shipped:__________ Laboratory ______________ Date shipped__________ Laboratory______________

Date shipped:__________ Laboratory ______________ Date shipped__________ Laboratory______________

****Notify the NWQL in advance of shipment of potentially hazardous samples—phone 1-866-ASK-NWQL or email LabLogin@usgs.gov**

Comments:

GROUNDWATER LEVEL NOTES

Station No.______________________ Field ID ____________

Station Name ______________________________________

Project No. ____________ Project Name_______________

Measurement made by: ______________________________

Signature __________________________ Date __________

WELL___ SPRING___ MONITOR___ SUPPLY___ OTHER______________

SUPPLY WELL PRIMARY USE: DOMESTIC__ PUBLIC SUPPLY__ IRRIGATION __ OTHER__________

Casing Material: ______________ **Altitude (land surface)** ________ **ft abv MSL(C16*)**

Measuring Point: ______ **ft abv blw LSD(C323*) MSL(C325*)**

Well Depth _______ ft abv blw LSD MSL MP

Casing/Well diameter (in)_______

Screened interval (ft): Top________ Bottom _________ft abv blw LSD MSL MP

Sampling condition (72006) pumping (8) flowing (4) static (n/a)

[see QWDATA User Manual for additional fixed-value codes]

Water Level: ____________ ft blw LSD (72019) ft blw MP (61055)

ft abv MSL (NGVD 29) (62610) ft abv MSL (NAVD 88) (62611)

Comments/Notes(C267) (256 character limit):

____

____

____

____

Depth to Water and Well Depth

	1ST	2ND	3RD (optional)
Time			
Hold (for DTW)			
□- Cut			
= DTW from MP (electric tape reading)			
– Measuring point (MP)			
= DTW from LSD			
Hold (for well depth)			
+ Length of tape leader			
= Well depth below MP			
– MP			
= Well depth below LSD			

WATER-LEVEL DATA FOR GWSI

DATE WATER LEVEL MEASURED (C235) __ __ - __ __ - __ __ __ __ TIME (C709) __ __ __ __
Month Day Year

TIME DATUM RELIABILITY CODE (C269)

E	K	T
estimated	known	transfered

TIME DATUM CODE (C402) __ __ __ __ __ __

WATER LEVEL (C237/241*/242) __ __ __ __ __.__ __

EQUIPMENT IDENTIFIER (C249) (26 character limit).

__ __

WATER LEVEL TYPE CODE (C243)

L	M	S
below land surface	below meas. pt.	sea level

WATER LEVEL DATUM (C245) (Mandatory if WL type=S)

NGVD 29	NAVD 88	| | | | | | | |
National Geodetic Vertical Datum Of	North American Vertical Datum Of 1988	Other (See GWSI manual for codes)

MP SEQUENCE NO. (C248) __ __ __
(Mandatory if WL type=M)

SITE STATUS FOR WATER LEVEL (C238)

A	B	C	D	E	F	G	H	I	J	M	N	O	P	R	S	T	V	W	X	Z
atmos. pressure	tide stage	ice	dry	recently flowing	flowing	nearby flowing	nearby recently	injector site	injector site	aquifer contact	measurement	obstruction	pumping	recently pumped	nearby pumping	nearby recently	foreign sub-	well des-	affected by surface	other

METHOD OF WATER-LEVEL MEASUREMENT(C239)

A	B	C	D	E	F	G	H	L	M	N	O	P	R	S	T	V	Z
airline	analog	calibrated airline	differential gps	estimated	transducer	pressure gage	calibrated pres. gage	geophysical logs	manometer	non-rec. gage	observed	acoustic pulse	reported	steel tape	electric tape	calibrated elec. tape	other

WATER LEVEL ACCURACY (C276)

0	1	2	9
foot	tenth	hundreth	not to nearest foot

SOURCE OF WATER-LEVEL DATA (C244)

A	D	G	L	M	O	R	S	Z
other gov't	driller's log	geologist	geophysical logs	memory	owner	other reported	reporting agency	other

PERSON MAKING MEASUREMENT (C246) (WATER-LEVEL PARTY) | | | | | |

MEASURING AGENCY (C247) (SOURCE) | | | | |

RECORD READY FOR WEB (C858)

Y	C	P	L
checked; ready for web display	not checked; no web display	proprietary; no web display	local use only; no web display

***Measuring Point Altitude (C325) or**
Measuring Point Height (C323) and Station Altitude (C16)
Are Required for Water Level (C241)

WELL PURGE LOG

FIELD ID ____________

Allowable Drawdown: ________ *ft* **Purge method:** STANDARD LOW-FLOW OTHER ______________________

START TIME:____________________ END TIME______________

Time	Water Level blw MP LSD ft	Draw-down ft	Well Yield gpm	Pumping Rate gpm	Water Temp °C	S C µs/cm	pH units	Dis-solved oxygen % sat	Turbidity ______ ______	______ ______	Comments [clarity, etc.]
											MEDIAN VALUES
											QUIESCENT pH
											FINAL FIELD MEASURE-

Well Volume (gal) = $V = 0.0408\ HD^2$ or Well Volume = $H \times F$

where:
V is volume of water in the well, in gallons
H is height of water column, in feet
D is inside Diameter of well, in inches
F is casing Volume Factor, in gallons per foot (see table)

H = Well depth - Static water level = __________ feet

Diameter, inside (D) = __________ inches

1 well volume (V) = __________ gallons

Parameter	Stability Criteria* (for more information see NFM Table 6.0-1)
pH	± 0.1 units (± 0.05 units if instrument display 2 or more digits to the right of the decimal) ± 0.3 if SC <~75µS/cm
Temperature (T)	± 0.2° C (thermistor)
Specific Conductivity (SC)	± 5%, of SC < 100 µS/cm ± 3%, for SC > 100 µS/cm
Dissolved Oxygen (DO)	± 0.2 mg/L
Turbidity (TU)	± 10%, for TU< 100: ambient TU is < 5 or most ground-water systems (visible TU > 5)

*allowable variation among 5 or more sequential field-measurement values

Purge Volume = (n)(V) = __________ **gallons [Actual =**__________ **gal]**

where:
n is number of well volumes to be removed during purging
V is volume of water in the well, in gallons

Q = estimated pumping rate = ______ gallons per minute

Approximate purge time = (purge volume)/Q = _______ minutes

	VOLUME FACTORS												
DIAMETER (in.)	1.0	1.5	2.0	3.0	4.0	4.5	5.0	6.0	8.0	10.0	12.0	24.0	36.0
CASING VOL. FACTOR (F)	0.04	0.09	0.16	0.37	0.65	0.83	1.02	1.47	2.61	4.08	5.88	23.5	52.9

Screened/Open Interval: TOP _________._____ ft blw LSD MSL

Bottom __________._____ ft blw LSD MSL

Depth to Top of Sampling Interval _______.____ft blw LSD MSL

Depth to Bottom of Sampling Interval _______.____ ft blw LSD MSL

Depth to set pump from MP (all units in feet except where noted) :

Distance to top of screen from LSD	
+ MP (- if MP below LS)	
– (7 to 10 x diameter (inches) of the well) [convert to feet]	
= Depth to pump intake from MP	

Depth to pump from LSD (all units in feet) :

– MP	
= Depth pump set from LSD MSL	

Notes/Calculations:

Calibrated by:______________________ Location:____________________ Station No. ______________
Date:______________ Time:____________

METER CALIBRATIONS and FIELD MEASUREMENTS

TEMPERATURE Meter make/model ________________ S/N ______________ Thermistor S/N ___________ Thermometer ID ________

Calibration criteria: ± 0.2 °C for thermistors

Local Meter

Lab Tested against NIST Thermometer/Thermistor? Y N Date: ________________ ±__________ °C

Measurement Location: SINGLE POINT AT ______ ft DEEP STREAMSIDE _____ FT FROM LEFT RIGHT BANK VERTICAL AVG/MEDIAN OF _____ PTS

Field Readings # 1 ______ #2 ______ #3 ______ #4 ______ #5 ______ **MEDIAN:**______ °C Method Code ______ Remark ___ ___ Qualifier __

SPECIFIC CONDUCTANCE Meter MAKE/MODEL ________________ S/N _____________ Sensor ID ________________

Sample: CONE SPLITTER CHURN SPLITTER SINGLE POINT AT ______ ft DEEP VERTICAL AVG. OF _______ POINTS

LOCAL METER ID:______________ AUTO TEMP COMPENSATED METER? Y N CORRECTION FACTOR APPLIES? Y N CORRECTION FACTOR:______

Std Value µS/cm	Std Temp	SC Before Adj.	SC After Adj.	Vendor Lot No.	NWIS Parameter Code (see last page)	NWIS* Lot No.	Expiration Date

Calibration Criteria: ± 5 % for SC ≤100 µS/cm or 3% for SC >100 µS/cm ***NWIS Lot Numbers are available at: http://wwwnwql.cr.usgs.gov/qas.shtml?ConductivityStds_home**

Field readings #1_____ **#2**_____ **#3**_____ **#4**_____ **#5** _____ **MEDIAN:** _______ **µS/cm** Method Code _______ Remark ___ ___ Qualifier ___

DISSOLVED OXYGEN Meter MAKE/MODEL ____________________________ S/N ________________

Sensor Type: Amperometric Luminescent Spectrophotometer Sensor ID ____________________ Local Meter ID____________

Calibration Method: Air-Saturated Water Water-Saturated Air

Sample: SINGLE POINT AT _______ ft DEEP VERTICAL AVG. OF _____ POINTS BOD BOTTLE OTHER____________ Stirrer Used? Y N

Calibration Temperature °C	Barometric Pressure mm Hg	DO Table Reading mg/L	Salinity Correction Factor	DO Before Adjustment mg/L	DO After Adjustment mg/L

Zero DO Check _________mg/lL Adj. to _________ mg/L Date: ____________

Thermister Check? Y N Date__________

Barometer Calibrated? N Y Date: ______________ Time: ________

Phase Degrees/Slope/Gain/Scale Factor (100%)______________(Zero)________

Calibration Criteria: ± 0.2 mg/L **DO saturation** ______ **%**

Field readings #1______ **#2**_____ **#3**_____ **#4**_____ **#5** _____ **MEDIAN:** _______ **mg/L** Method Code _______ Remark ___ ___ Qualifier ____

pH Meter MAKE/MODEL ________________ S/N ______________ Electrode ID ______________ Type: GEL LIQUID OTHER ________

Sample: FILTERED UNFILTERED CONE CHURN SPLITTER SINGLE POINT AT _______ ft DEEP VERTICAL AVG. OF ____ POINTS

TEMPERATURE CORRECTION FACTORS APPLIED TO BUFFERS? Y N

pH BUFFER	BUFFER TEMP	THEORETICAL pH FROM TABLE	pH BEFORE ADJ.	pH AFTER ADJ.	SLOPE	MILLIVOLTS
pH 7						
pH ___						
CHECK pH ___						

pH Buffer	Vendor Lot No.	NWIS* Lot No.	Expiration Date
pH 7 (99173)			
pH 10 (99171)			
pH 4 (99172)			

Calibration Criteria: ± 0.1 pH units, ± 0.3 if SC <75us/cm ***NWIS Lot Numbers are available at: http://wwwnwql.cr.usgs.gov/qas.shtml?Buffers_home**
Millivolts: pH 7 –10 to +10 , pH4 +165 to +195 mV, pH 10 –165 to –195 mV
Slope Acceptance Criteria: 95% to 102%

Field Readings #1_________ **#2**_________ **#3**_________ **#4**_________ **#5** _________ **MEDIAN:** _______ **Units** Remark ___ ___ Qualifier ___

FIELD ID ______________

TURBIDITY Meter make/model ______________ S/N ____________ Type: turbidimeter submersible spectrophotometer

Sample: pump discharge line flow-thru chamber single point at ______ ft blw LSD MSL MP Sensor ID ____________

Sample: Collection Time: ________ Measurement Time: ________ Measurement: In-situ/On-site Vehicle Office lab NWQL Other __________

Sample diluted? Y N Vol. of dilution water ________ mL Sample volume ________ mL

	Lot Number or Date Prepared	Expiration Date	Concentration ______ (units)	Calibration Temperature °C	Initial instrument reading	Reading after adjustment
Stock Turbidity Standard						
Zero Standard *(DIW)*						
Standard 1						
Standard 2						
Standard 3						

TURBIDITY VALUE = $A \times (B+C) / C$

where:

A= TURBIDITY VALUE IN DILUTED SAMPLE

B= VOLUME OF DILUTION WATER, mL

C= SAMPLE VOLUME, mL

Calibration Criteria:

$\leq$ 100 Turbidity units	± 0.5 turbidity units or ± 5% of the measured Value, whichever Is greater
> 100 Turbidity units	± 10%

Field Readings #1 __________ #2 __________ #3 __________ #4 __________ #5 __________

MEDIAN ________ **Parameter Code** __________ FNU NTU NTRU FNMU FNRU FAU FBU AU METHOD CODE __________ Remark ___ ___ Qualifier ___ ___

QUALITY-CONTROL INFORMATION

PRESERVATIVE, BLANK WATER and SPIKE NWIS LOT NUMBERS

NWIS lot numbers are available at: http://wwwnwql.cr.usgs.gov/qas.shtml?nfssqa_certificates

Description	Parameter Code	ExpirationDate	Manufacturer Lot Number	NWIS Lot Number
4.5N H_2SO_4 (NUTRIENTS AND DOC)	99156			
7.5N-7.7N HNO_3(METALS&CATIONS)	99159			
6N HCl (Mercury)	99158			
1:1 HCl (VOC)	99157			
18N H_2SO_4 (COD and Phenol)	99155			
Inorganic Blank Water	99201			
Organic Blank Water	99203			
VOC/Organic Blank Water	99205			
Spike	99104			

Filter Lot Numbers

Filter descriptions with parameter codes require NWIS LOT NUMBERS available at http://wwwnwql.cr.usgs.gov/qas.shtml?filters_home

Filter Type	Pore Size (microns)	Parameter Code	Manufacturer's Lot Number	NWIS Lot Number
Capsule	0.45	**99206**		
Disc	0.45	**99206**		
142 mm GFF (organics)	0.70			
Syringe (organics)	0.70	99207		
25 mm GFF (organic carbon)	0.70			
142 mm membrane (inorganics)	0.45			

QC SAMPLES

Sample Type	NWIS Record No.	Sample Type	NWIS Record No.	Sample Type	NWIS Record No.
Equip Blank _____	__________	Sequential _____	__________	Trip Blank _________	__________
Field Blank _____	__________	Spike _____	__________	Other _________	__________
Split _____	__________	Concurrent _____	__________	Other _________	__________

NWQL Schedules/lab codes (QC Samples) __________ __________ __________ __________ __________ __________

ALKALINITY/ANC

BEGINNING H_2O TEMP. _________ °C

Specific Conductance___________µS/cm

BEGINNING H_2O TEMP. _________ °C

Specific Conductance___________µS/cm

pH	ΔpH	VOL ACID DC OR mL	ΔVOL ACID DC OR mL	ΔpH / ΔVOL ACID	pH	ΔpH	VOL ACID DC OR mL	ΔVOL ACID DC OR mL	ΔpH / ΔVOL ACID

Use the Alkalinity Calculator at: **http://or.water.usgs.gov/alk** or PCFF

Alkalinity/ANC pH Meter Calibration

Meter make/model:	S/N	
Calibration Location:		
Electrode No.		
Electrode type GEL LIQUID		
	pH 7	pH ____
BUFFER TEMPERATURE		
THEORETICAL FROM TABLE		
pH BEFORE ADJUSTMENT		
pH AFTER ADJUSTMENT		
SLOPE		
MILLIVOLTS		
MANUFACTURER LOT NUMBER		
NWIS LOT NUMBER		

End H_2O temp. _________ **°C**

Specific Conductance___________**µS/cm**

End H_2O temp. _________ **°C**

Specific Conductance___________**µS/cm**

FIRST TITRATION

DATE ______________ INITIALS _______________

BEGIN TIME____________ END TIME____________

ALKALINITY/ANC ______________ mg/L AS $CaCO_3$

BICARBONATE _________________mg/L AS HCO_3^-

CARBONATE __________________mg/L AS CO_3^{2-}

HYDROXIDE __________________mg/L AS OH^-

ACID: 1.6N 0.16N 0.01639N OTHER:_______

ACID LOT NO. ________EXPIRATION DATE __________

CORRECTION FACTOR: 1.00 1.01

ACID DELIVERY: DIGITAL COUNTER BURET

SAMPLE VOLUME: ________mL FILTERED UNFILTERED

METHOD: INFLECTION POINT GRAN

SECOND TITRATION

DATE ______________ INITIALS _______________

BEGIN TIME____________ END TIME____________

ALKALINITY/ANC ______________ mg/L AS $CaCO_3$

BICARBONATE _________________mg/L AS HCO_3^-

CARBONATE __________________mg/L AS CO_3^{2-}

HYDROXIDE __________________mg/L AS OH^-

ACID: 1.6N 0.16N 0.01639N OTHER:_______

ACID LOT NO. ________EXPIRATION DATE __________

CORRECTION FACTOR: 1.00 1.01

ACID DELIVERY: DIGITAL COUNTER BURET

SAMPLE VOLUME: ________mL FILTERED UNFILTERED

METHOD: INFLECTION POINT GRAN

Field titration by:________________

Checked by:______________

REFERENCE LIST FOR CODES USED ON THIS FORM

Sample Medium Codes

WG	Regular Ground water
WGQ	Quality-control sample (Replicate or Spike)
OAQ	Blank

The complete list of fixed-value codes can be found online at: http://nwis.usgs.gov/nwisdocs4_10/gw/QW-AppxB.pdf

Time Datum Codes

Time Zone	Std Time Code	UTC Offset (hours)	Daylight Time Code	UTC Offset (hours)
Hawaii-Aleutian	HST	-10	HDT	-9
Alaska	AKST	-9	AKDT	-8
Pacific	PST	-8	PDT	-7
Mountain	MST	-7	MDT	-6
Central	CST	-6	CDT	-5
Eastern	EST	-5	EDT	-4
Atlantic	AST	-4	ADT	-3

71999 Sample purpose

10	Routine
15	NAWQA
50	GW Network
110	Seepage Study
120	Irrigation Effects
130	Recharge
140	Injection

Sample Type Code

9	Regular
7	Replicate
2	Blank
1	Spike
3	Reference
B	Other QA
H	Composite

Value Qualifiers

e	see field comment
f	sample field preparation problem
k	counts outside the acceptable range

Null-value Qualifiers

e	required equipment not functional or available
f	sample discarded; improper filter used
o	insufficient amount of sample
p	sample discarded; improper preservation
q	sample discarded; holding time exceeded
r	sample ruined in preparation

50280 Purpose of site visit

2001	Primary (primary samples should not exist for a site for more than one date per HIP, and the primary sampling date generally has the highest number of NAWQA analytes)
2002	Supplemental (to fill in missing schedules not sampled or lost)
2003	Temporal characterization (for previously sampled schedules; includes LIP and seasonal samples)
2004	Resample (to verify questionable concentrations in primary sample)
2098	Ground-water quality control
2099	Other (ground-water related samples with medium code other than "6", such as soil samples or core material)

82398 Sampling method

4010	Thief sampler
4020	Open-top bailer
4025	Double-valve bailer
4030	Suction pump
4040	Submersible pump
4045	Submersible multiple impeller (turbine) pump
4050	Squeeze pump
4060	Gas reciprocating pump
4070	Gas lift
4080	Peristaltic pump
4090	Jet pump
4100	Flowing well
4110	Resin trap collector
8010	Other

84164 Sampler type

4010	Thief Sampler
4020	Open-top Bailer
4025	Double-valve Bailer
4030	Suction Pump
4035	Submersible Centrifugal Pump
4040	Submersible Positive-pressure Pump
4041	Submersible Helical Rotor Pump
4045	Submersible Gear Pump
4050	Bladder Pump
4060	Gas Reciprocating Pump
4070	Gas Lift
4075	Submersible Piston Pump
4080	Peristaltic Pump
4090	Jet pump
4095	Line-Shaft Turbine Pump
4100	Flowing Well
8010	Other

72006 Sampling Condition

0.01	The site was dry (no water level is recorded)
0.02	The site had been flowing recently
0.03	The site was flowing, head could not be measured
0.04	A nearby site that taps the Aquifer was flowing
0.05	Nearby site tapping same Aquifer had been flowing recently
0.06	Injector site
0.07	Injector site monitor
0.08	Measurement discontinued
0.09	Obstruction encountered in well above water surface
0.10	The site was being pumped
0.11	The site had been pumped recently
0.12	Nearby site tapping the same Aquifer was being pumped
0.13	Nearby site tapping the Same Aquifer was pumped recently
0.14	Foreign substance present on the surface of the water
0.16	Water level affected by stage in nearby site
0.17	Other conditions affecting the measured water level
2	Undesignated

4	Flowing	23	Flowing to Pit
6	Flowing on gas lift	24	Water Flooding
8	Pumping	25	Jetting
10	Open hole	30	Seeping
18	Producing	31	Nearby well pumping
19	Circulating	32	Nearby well taking water
22	Lifting	33	Well taking water

71875 Hydrogen Sulfide Odor

Value
\# none entered (null)

Remark Code	Method Code
M detect	U un-acidified sample
U non-detect	V acidified sample

00003 Sampling depth, ft
78890 Sampling depth, ft blw msl
00059 Flow rate, instantaneous, gallons per minute
72004 Pump or flow period prior to sampling, minutes

Water Level
61055 Water level, depth below measuring point, feet
62610 Ground-water level above NGVD 1929, feet
62611 Ground-water level above NAVD 1988, feet
72019 Depth to water level, feet below land surface

NWIS Lot Number Parameter Codes* for Conductance Standards

Parameter Code	Standard Value µS/cm, KCl
99160	50
99161	100
99162	250
99163	500
99164	750
99165	1000
99166	2500
99167	5000
99168	10,000
99169	25,000
99170	50,000

Dissolved Oxygen

AZIDE	Azide-modified Winkler	INDIGO	Sepctrophotometer, indigo carmine
INDKT	Field Kit, ndigo carmine, visual	LUMIN	Luminscence sensor
MEMB2	Amperometric, Membrane (DODEC)	MEMBR	Ampermetric, Membrand electrode
RHODA	Field Kit, Rhodazine-D, visual	SPC10	Spectrophotometer, Rhodazine-D

Parameter and method codes for field measurements: http://water.usgs.gov/usgs/owq/Forms.html

***NWIS Lot numbers and Certificates of Analysis: http://wwwnwql.cr.usgs.gov/qas.shtml?nfssqa_certificates**

National Field Manual: http://water.usgs.gov/owq/FieldManual/

Alkalinity Calculator, Alkalinity/ANC parameter and method codes: http://or.water.usgs.gov/alk/reporting.html

Appendix V – EPA Region 8 Laboratory Chain of Custody Form

US ENVIRONMENTAL PROTECTION AGENCY REGION 8 - Chain Of Custody

Region 8 Laboratory
16194 W. 45th Drive
Golden, CO 80403
Fax: 303-312-7800

PROJECT NAME:

LSR #:

SAMPLER(S):
Name: **Signature:** **Date:**
Name: **Signature:** **Date:**

						ANALYSIS								Comments
STATION ID.	STATION DESCRIPTION + NOTES	DATE	TIME	MATRIX	PRESERVATIVE	Analysis	Analysis 2						NUMBER OF CONTAINERS	LAB ID.
#1 (used in lab report)	More information for your use			W	9	X							1	
				W	9		X						1	
#2	More information for your use			W	9	X							1	
				W	9		X						1	
#3	More information for your use			W	9	X							1	
				W	9		X						1	

1-Relinquished By: Date/Time	2-Relinquished By: Date/Time	3-Relinquished By: Date/Time
1-Received By: Date/Time	2-Received By: Date/Time	3-Received By: Date/Time

PRESERVATIVES 1- HNO_3 2-H_3PO_4 3- H_2SO_4 4-HCl
5- $Na_2S_2O_3$ 6- Ice 7-Dry Ice 8- NaOH 9- Other:___none_________

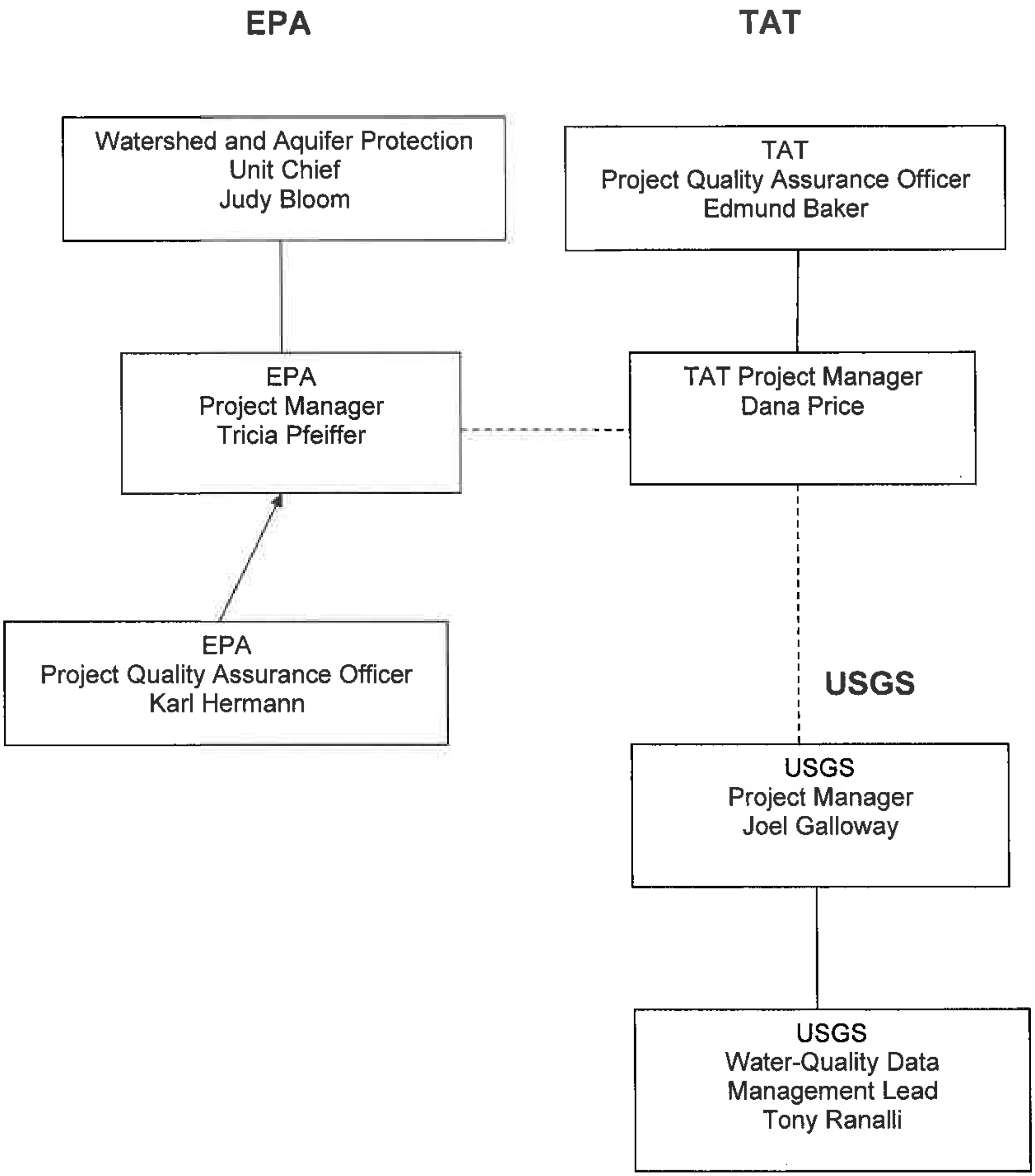
MATRIX
W- Water S- Soil Sed- Sediment Bio- Bio Tissue O- Other__________

Cooler Temperature =

Page ____ of ____
EPA R8 Lab Form No.: GENLF-001 Rev. 1.0

Appendix VI – Organization Chart

Organization Chart



Appendix VII – USACE Sampling Locations

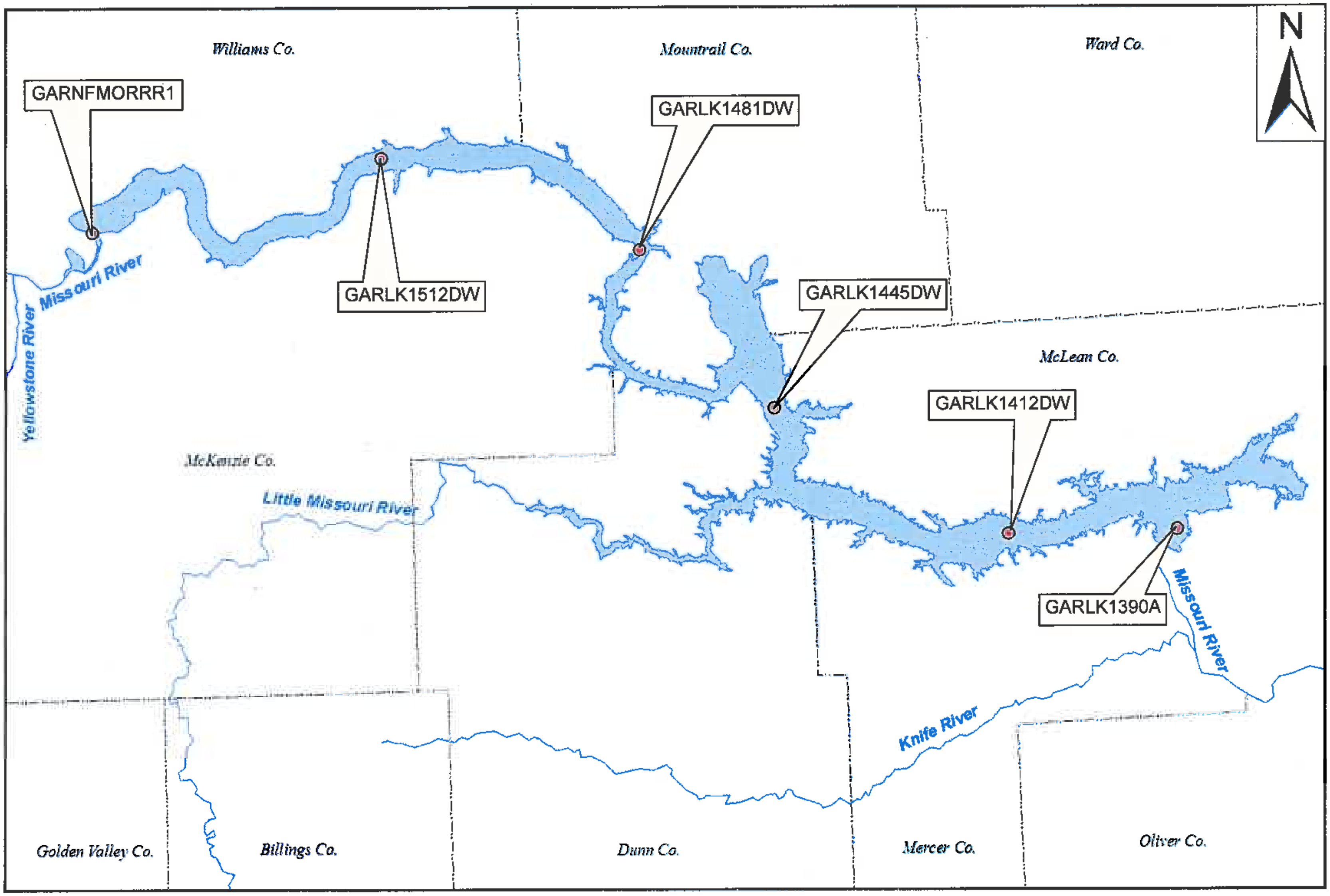
Monitoring Site Locations and Numbers.

Project	Sampling Site Location	Site Number	Latitude	Longitude
Fort Peck	Near Dam	FTPLK1772A	47° 59' 03.0"	106° 25' 09.2"
	Hell Creek	FTPLK1805DW	47° 40' 48.7"	106° 52' 45.4"
	Rock Creek	FTPLKBDCA02	47° 44' 40.1"	106° 17' 42.9"
	Inflow (Missouri River)	FTPNFMORR1	US Highway 191 Bridge Crossing (Landusky, MT)	
Garrison	Near Dam	GARLK1390A	47° 30' 56.0"	101° 22' 17.4"
	Beulah Bay	GARLK1412DW	47° 31' 15.4"	101° 45' 44.6"
	Deepwater Bay	GARLK1445DW	47° 42' 56.7"	102° 15' 29.6"
	New Town	GARLK1481DW	47° 59' 22.5"	102° 33' 46.0"
	White Tail Bay	GARLK1512DW	48° 08' 27.5"	103° 07' 52.6"
	Inflow (Missouri River)	GARNFMORRR1	US Highway 85 Bridge Crossing (Williston, ND)	
Oahe	Near Dam	OAHLK1073A	44° 27' 45.6"	100° 25' 18.5"
	Cheyenne River	OAHLK1110DW	44° 46' 15.3"	100° 43' 02.3"
	Whitlocks Bay	OAHLK1153DW	45° 01' 51.3"	100° 16' 43.8"
	Mobridge	OAHLK1196DW	45° 32' 29.2"	100° 29' 08.8"
	Beaver Creek	OAHLK1256DW	46° 15' 03.6"	100° 33' 20.9"
	Inflow (Missouri River)	OAHNFMORR1	Interstate 94 Bridge Crossing (Bismarck, ND)	
	Inflow (Cheyenne River)	OAHNFCHYR1	SD Highway 63 Bridge Crossing	
Big Bend	Near Dam	BBDLK0987A	44° 02' 21.6"	99° 26' 54.9"
	Iron Nation	BBDLK1020DW	44° 06' 51.9"	99° 41' 58.5"
	Antelope Creek	BBDLK1055DW	44° 18' 37.3"	100° 06' 04.3"
Fort Randall	Near Dam	FTRLK0880A	43° 03' 28.9"	98° 34' 37.8"
	Platte Creek	FTRLK0911DW	43° 16' 47.0"	99° 00' 40.6"
	Elm Creek	FTRLK0940DW	43° 33' 54.8"	99° 19' 37.4"
	Chamberlain	FTRLK0968DW	43° 49' 50.9"	99° 19' 36.6"
Gavins Point	Near Dam	GPTLK0811A	42° 52' 10.4"	97° 29' 47.3"
	Bloomfield	GPTLK0819DW	42° 51' 0.6"	97° 38' 34.2"
	Charley Creek	GPTLK0825DW	42° 50' 26.8"	97° 45' 19.6"
	Inflow (Missouri River)	GPTNFMORR1	Boat ramp at Running Water, SD	

Monitoring Sites, Sample Types, and Collection Frequency

Reservoir monitoring sites will be located where the greatest water depth occurs in the region of the reservoir being sampled. This will be over the old Missouri River channel that is now submerged by the mainstem reservoir. One of the sites at Fort Peck Reservoir will be located in the old Big Dry Creek channel that is now submerged. Water quality grab samples will be collected at two depths: near-surface and near-bottom. The near-surface sample will be collected at ½ the measured Secchi depth and the near-bottom sample will be collected within 1-meter of the reservoir bottom. Profile measurements will also be taken from the reservoir surface to the bottom in 1-meter increments. The reservoir sites are to be monitored monthly from May through September/October, and the monthly monitoring is to be separated by at least 21 days but not more than 35 days. Depth-profiles will be measured at Fort Peck, Garrison and Oahe during October if turn-over has not completely occurred at the reservoir when profiles are measured in September.

Inflow near-surface grab samples will be collected from the Missouri and Cheyenne Rivers near the bank in an area of faster current. The inflow sites will be sampled monthly from April through October.



Water quality monitoring site locations and numbers for Garrison Project

Appendix VIII – Additional Support Equipment

Equipment

tools	squirt bottles
steel tape and electric tape for WL measurement	HACH 2100N turbidimeter
field notes	peristaltic pump
faucet connector and disposable adapters	coolers
back-flow preventer	fittings
TFE tubing	Grundfos Rediflo2 sampling pump with TFE tubing
TFE tubing manifold	teflon standpipe for pump decon
processing chamber	HACH field alkalinity kit
chamber bags	Hach pH meter for alkalinity
clips for chamber bag	generator and fuel for pump
gloves	TFE bottle for DH-81 -SW only
tubing -small teflon for VOC collection	TFE nozzle and adapter for DH-81 - SW only
tubing c-flex	poly churn -SW only
YSI or Hydrolab multisonde	VOC sampler -SW only
flow-thru cell for multisonde	US DH-81 sampler -SW only
peristaltic pump	barometer

Appendix IX – Qualifiers

A	Compound not analyzed
ANR	Analyte Not Requested
B	Detection in blank:
BH	Detection in blank. Holding time exceeded.
BU	Detection in blank. Not Detected: The analyte was analyzed for, but was not detected at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method.
C	Presence of compound may be due to contamination of sample during laboratory processing
D	Contract Required Quantitation Limit (CRQL) not met due to sample matrix interference, dilution required.
DB	Contract Required Quantitation Limit (CRQL) not met due to sample matrix interference, dilution required. Detection in blank.
DH	Contract Required Quantitation Limit (CRQL) not met due to sample matrix interference, dilution required. Holding time exceeded
DJ	Contract Required Quantitation Limit (CRQL) not met due to sample matrix interference, dilution required. Estimated:The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample.
DR	Contract Required Quantitation Limit (CRQL) not met due to sample matrix interference, dilution required. Rejected: The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.
DU	Contract Required Quantitation Limit (CRQL) ... Not Detected: The analyte was analyzed for, but was not detected at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method.

E	Concentration of analyte being analyzed exceeded calibration range of instrument.
EE	Identifies compounds whose concentration exceed the calibration range addition of the instrument for that specific analysis.
EFAI	Equipment failure
F	Estimated value: compound failed intitial calibration check (CCC) or QC criteria
FEQ	Field Equipment Questionable
H	Holding time exceeded:
HBJ	Holding time exceeded. Detection in blank. Estimated: The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample.
HL	Holding time exceeded. Lowest available reporting limit for the analytical method used.
HLBL	high labeled compound recovery in sample, estimated value, estimated value
HMSR	high matrix spike recovery, potential high bias
HNRO	high native analyte recovery in OPR (or LCS), potential high bias
HSSR	high surrogate spike recovery, potential high bias
HVER	high calibration verification standard recovery, estimated value
I	Estimated value; compound failed initial calibration value
ITNA	Incubation time not attained
ITNM	Incubation temperature not maintained
J	Estimated: The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample.
J+	Estimated: The analyte was positively identified and the associated numerical value... +++.
J-MI	Approximate value due to matrix interference
J-QC	Approximate value due to quality control problems
J-R	Approximate value result is below the reporting level but greater than the method detection limit

JB	Estimated: The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample. Detection in blank.
JH	Estimated: The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample. Holding time exceeded.
JHTF	Holding Time Exceeded in the Field
JL	Estimated: The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample. Lowest available reporting limit for the analytical method used.
K	Value below the detection Limit. For BOD: depletion is less than 1.0
KK	True bacterial concentration is assumed to be less than the reported value.
L	Lowest available reporting limit for the analytical method used.
LL	True bacterial concentration is assumed to be greater than the reported value.
LLBL	low labeled compound recovery in sample, estimated value
LLRO	low labeled compound recovery in the OPR (or LCS), estimated value
LMSR	low matrix spike recovery, potential low bias
LNRO	low native analyte recovery in OPR (or LCS), potential low bias
LOPR	low OPR (or LCS) recovery, potential low bias
LVER	low calibration verification standard recovery, potential low bias
MTRX	possible matrix interference, estimated value
N	Presumptive evidence of a nontarget compound
NA	Not Applicable
NAP	No Analysis Possible
NCNF	not confirmed or not found, estimated value
NLBL	no labeled compound recovery in sample, rejected
NLRO	no labeled compound recovery in OPR (or LCS), rejected
NMSR	no matrix spike recovery, rejected

NNRO	no native analyte recovery in OPR (or LCS), rejected
NOPR	no OPR (or LCS) recovery, rejected
NRP	No result possible
NVER	no calibration verification standard recovery, rejected
QNS	Quantity Not Sufficient
R	Rejected: The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.
RMAX	result is a maximum value
RNAF	result no affected by noted QC issue
RNF2	results of 2 columns not within factor of 2, estimated value
RNON	result reported as non-detect due to blank contamination
RPDX	RPD is MS/MSD pair exceeds criterion, estimated value
U	Not Detected: The analyte was analyzed for, but was not detected at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method.
UH	Not Detected: The analyte was analyzed for, but was not detected at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method. Holding time exceeded.
UJ	Not Detected/Estimated: The analyte was not detected at a level greater than or equal to the adjusted CRQL or the reported adjusted CRQL is approximate and may be inaccurate or imprecise.

EPA REGION 8 QA DOCUMENT REVIEW CROSSWALK

QAPP/FSP/SAP for: *(check appropriate box)*		Entity *(grantee, contract, EPA AO, EPA Program, Other)* ~~Tricia Pfeiffer, EPA Region 8, Watershed and Aquifer Protection Unit~~ Three Affiliated Tribe	**Regulatory Authority** **and/or** **Funding Mechanism**	**_X_40 CFR 31 for Grants** **___48 CFR Part 46 for Contracts** **___ Interagency Agreement** **___ EPA Administrative Order** **___ EPA Program Funding** **___ EPA Program Regulation** **_~~X~~_ EPA CIO 2105**
X	**GRANTEE**			
	CONTRACTOR			
~~X~~	**EPA**			
	Other			
Document Title *[Note: Title will be repeated in Header]*		QAPP for the Ambient Monitoring of Surface Water and Groundwater Quality		
QAPP/FSP/SAP Preparer		~~Tricia Pfeiffer~~ Dana Price		
Period of Performance *(of QAPP/FSP/SAP)*		**2014-2019**	**Date Submitted for Review**	03/03/2014
EPA Project Officer **EPA Project Manager**		Blake Huff Tricia Pfeiffer, EPA Project Manager	**PO Phone #** **PM Phone #**	303-312-6718 303-312-6271
QA Program Reviewer or Approving Official		Jeff Pritt (303-313-7077)	**Date of Review**	Version 2014.02.14 was reviewed 4/4/2014 Version 2014.02.14 submitted on 4/25/2014 was reviewed 4/28/2014 Third review 4/30/2014

Documents to Review:

1. QAPP written by Grantee or EPA must also include for review:
   Work Plan(WP) / Statement of Work (SOW) / Program Plan (PP) / Research Proposal (RP)

2. QAPP written by Contractor must also include for review:
   a) Copy of signed QARF for Task Order
   b) Copy of Task Order SOW
   c) Made available hard or electronic copy of approved QMP
   d) If QMP not approved, provide Contract SOW

3. For a Field Sampling Plan (FSP) or Sampling & Analyses Plan (SAP), the Project QAPP must also be provided.
   OR
   The FSP or SAP must be clearly identified as a stand-alone QA document and must contain all QAPP required elements (Project Management, Data Generation/Acquisition, Assessment and Oversight, and Data Validation and Usability).

Documents Submitted for QAPP Review:

1. QA Document(s) submitted for review:

QA Document	**Document Date**	**Document Stand-alone**	**Document with QAPP**
QAPP	~~03/03/2014~~ **Feb. 2014**	Yes ~~N/A~~	
FSP	**N/A**	No ~~N/A~~	N/A
SAP	**N/A**	No ~~N/A~~	N/A
SOP(s)	**Included**		N/A

2. WP/SOW/TO/PP/RP Date __provided without a date__ ~~N/A~~_____
WP/SOW/TO/RP Performance Period _10-1-2013 to 9-30-2014___ ~~N/A~~________

3. QA document consistent with the:
WP/SOW/PP for grants? _Yes ~~N/A~~
SOW/TO for contracts? _No ~~N/A~~

4. QARF signed by R8 QAM N/A
Funding Mechanism _Grant ~~N/A~~
Amount _$164,442__ ~~Region 8 Program~~

Summary of Comments *(highlight significant concerns/issues)*:

1. The QAPP needs to be clear that the grantee (Three Affiliated Tribes) is responsible for all project activities and that USGS is directed by and advising the Tribe. See individual comments in the crosswalk. **Resolved**
2. The QAPP needs to be clear that EPA advises the Tribe. See individual comments in the crosswalk. **Resolved**
3. The QAPP needs to state all data collected by USGS directly or from other sources in support of the Tribal Water Quality Program and for preparation of the Scientific Investigations Report is provided to the Tribe in a usable electronic format to support the Tribe's water quality monitoring goals. (See comment A6.a) Resolved
4. The QAPP needs to have more specific description, including existing data sources for establishing the rationale for selecting the 24 targeted variable sites. (See comment B1.c) **Resolved**
5. **This multi-year QAPP needs an annual update and review by the Tribe and submitted to EPA for approval. (See comments C1.a and C2.a) Resolved**
6. **The** Three Affiliated Tribe **must address the comments in the Summary of Comments, as well as those identified in the Comment section(s) that includes a "Response (date)" and Resolved (date)". Partially Resolved- see comments in** Green. Resolved.

Element	Acceptable *Yes/No/NA*	Page/ Section	Comments
A. Project Management			
A1. Title and Approval Sheet			
a. Contains project title	Y	A1, 2	
b. Date and revision number line (for when needed)	Y	A1,.2	
c. Indicates organization's name	Y	A1, 2	
d. Date and signature line for organization's project manager	Y	A1,.2	
e. Date and signature line for organization's QA manager	Y	A1, 2	
f. Other date and signatures lines, as needed	Y	A1, 2	
A2. Table of Contents			
a. Lists QA Project Plan information sections	Y	A2, 3	
b. Document control information indicated	Y	A2, 3	
A3. Distribution List			
Includes all individuals who are to receive a copy of the QA Project Plan and identifies their organization	Y	A3, 4-5	
A4. Project/Task Organization			
a. Identifies key individuals involved in all major aspects of the project, including contractors	~~N~~Y	A4, 5-6	Please add the word "**maintain**" to "preparing[**, maintaining**] and implementing the QAPP" in the fourth sentence of Dana Price's

b. Discusses their responsibilities			responsibilities. **Response (date):** pg 5: A4.1: Paragraph 1, line 5 **Resolved (date): Resolved 4/28/2014** Please add to the second sentence from Edmund Baker's environmental director's responsibilities to include approving the QAPP **"for Three Affiliated Tribes."** **Response (date):** pg 5: A4.1: Paragraph 2, line 3 **Resolved (date): Resolved 4/28/2014** Please clarify the second sentence for Tony Ranalli's roles and responsibilities **to "assisting the TAT Water Quality Coordinator (Dana Price) with quality assuring and interpreting all data collected for TAT and ensuring all data collected are provided electronically and in reports to Three Affiliated Tribes."** **Response (date):** pg 5: A4.1: Paragraph 4, line 3-4 **Resolved (date): Resolved 4/28/2014** Please use a second heading in Section A4 called "EPA Roles and Responsibilities to clearly indicate EPA is not involved directly with the TAT project organization **Response (date):** pg 5-6 A4.1 & A4.2 Titles added **Resolved (date): Resolved 4/28/2014**
c. Project QA Manager position indicates independence from unit generating data	Y	A4, 5	
d. Identifies individual responsible for maintaining the official, approved QA Project Plan	~~N~~ Y	A4, 5	See first comment in A4.a/b above. **Response (date):** **Resolved (date): Resolved 4/28/2014**
e. Organizational chart shows lines of authority and reporting responsibilities	Y	App. I, 51	Please include the level II QA staff member in the organization chart and the distribution list. Note: this individual is identified to have key responsibilities in the data verification and validation process. (See Section D1 on page 31) **Response (date):** removed sent (language from previous QAPP) **Resolved (date): Resolved 4/28/2014**
A5. Problem Definition/Background			
a. States decision(s) to be made, actions to be taken, or outcomes expected from the information to be obtained	Y	A5, 6-7	
b. Clearly explains the reason (site background or historical context) for initiating this project	Y	A5, 6-7	Revised last sentence of 1st paragraph (pg7)

c. Identifies regulatory information, applicable criteria, action limits, etc. necessary to the project		A5, 6-7; A6, 7-8, A7, 12	
A6. Project/Task Description			
a. Summarizes work to be performed, for example, measurements to be made, data files to be obtained, etc., that support the project's goals	~~N~~ Y	A6, 7-12	Please remove "if possible" from the statement in Task I on page 12 and be clear that all data collected by USGS directly or from other sources for preparation of the Scientific Investigations Report is provided to the Tribe in a usable electronic format to support the Tribe's water quality monitoring goals. **Response (date):**removed (pg 12) **Resolved (date): Resolved 4/28/2014**
b. Provides work schedule indicating critical project points, e.g., start and completion dates for activities such as sampling, analysis, data or file reviews, and assessments	~~N~~ Y	A6, 7-12	Please remove "under a cooperative agreement" form the last sentence of the second paragraph in Section A6. **Response (date):**removed (pg8) **Resolved (date): Resolved 4/28/2014** Please state that EPA Region 8 Laboratory will provide analytical data to the Tribe and to USGS. Note: The Tribe and USGS will do the data review. **Response (date):**pg10: last paragraph, 1st set revised **Resolved (date): Resolved 4/28/2014**
c. Details geographical locations to be studied, including maps where possible	Y	Table 1-2, 36-37, Figures 2-3, 34-35	
d. Discusses resource and time constraints, if applicable	Y	A5, 6-7	
A7. Quality Objectives and Criteria			
a. Identifies - performance/measurement criteria for all information to be collected and acceptance criteria for information obtained from previous studies, - including project action limits and laboratory detection limits and - range of anticipated concentrations of each parameter of interest	~~N~~ Y	A7, 12-14	For reference, please include the citation of the procedures and criteria [Rantz and others 1982] USGS will use for stream discharge measurement to last sentence of the first paragraph on page 13. The easiest way to include a citation in the text of the QAPP is to look in the Literature Cited which starts on page 33 and copy the first part of the reference up to and including the year and paste within parenthesis. **Response (date):**pg 13: last paragraph, last set revised **Resolved (date): Resolved 4/28/2014**
b. Discusses precision	Y	B1, 18	
c. Addresses bias	Y	B1, 18	

d. Discusses representativeness	Y	B1, 19	
e. Identifies the need for completeness	Y	B1, 19	
f. Describes the need for comparability	Y	B1, 19	
g. Discusses desired method sensitivity	Y	B1, 17	
A8. Special Training/Certifications			
a. Identifies any project personnel specialized training or certifications	Y	A8, 15	
b. Discusses how this training will be provided	Y	A8, 15	
c. Indicates personnel responsible for assuring training/certifications are satisfied	~~N~~ Y	A8, 15	The QAPP needs to state the Tribal Project Manager (Water Quality Coordinator) is responsible for documenting and maintaining the training records in the project document and records in the first paragraph in Section A8 on page. **Response (date):**pg15: A8 1st paragraph last sent revised **Resolved (date): Resolved 4/28/2014**
d. identifies where this information is documented	~~N~~ Y	A9, 15	
A9. Documentation and Records			
a. Identifies report format and summarizes all data report package information	Y	A9, 15-16	
b. Lists all other project documents, records, and electronic files that will be produced	~~N~~ Y	A9, 15-16	See comment A8.d above.
c. Identifies where project information should be kept and for how long	Y	A9, 15-16	
d. Discusses back up plans for records stored electronically	Y	B10, 28-29	
e. States how individuals identified in A3 will receive the most current copy of the approved QA Project Plan, identifying the individual responsible for this	Y	A9, 15-16	
B. Data Generation/Acquisition			
B1. Sampling Process Design (Experimental Design)			
a. Describes and justifies design strategy, indicating size of the area, volume, or time period to be represented by a sample	Y	B1, 17	
b. Details the type and total number of sample types/matrix or test runs/trials expected and needed	Y	A6, 8-12, Appendix II	Note that the QAPP will need to be updated with the location information for the actual springs sampled.

c. Indicates where samples should be taken, how sites will be identified/located	~~N~~ Y	A6, 7-12; B1-2, 17-21	The QAPP needs to be more specific about how the 24 targeted variable sites will be selected. Please include description of areas with existing knowledge of spills (including data sources or web links) in the QAPP and describe the rationale for selection of the targeted variable sites. It is not clear if the targeted variable sampling sites scheduled for the June sampling event are the same or different sites for the August sampling event. **Response (date):**revised pg 7, added USACE sampling assistance and App VII; pg 11 revised Task 1, Drainage sampling 2nd paragraph, 2nd sent **Resolved (date):** USACE will be collecting samples according to this QAPP; therefore USACE needs to be added to the distribution list. Response: Resolved: Resolved 4/30/2014
d. Discusses what to do if sampling sites become inaccessible	Y	B1, 11	
e. Identifies project activity schedules such as each sampling event, times samples should be sent to the laboratory, etc.	Y	A6, 9-12; A9, 15-16; B1, 17, Table 3	
f. Specifies what information is critical and what is for informational purposes only	Y	B1, 17-19	
g. Identifies sources of variability and how this variability should be reconciled with project information	Y	B1, 18-19 B5,24+26-27 D3, 32	
B2. Sampling Methods			
a. Identifies all sampling SOPs by number, date, and regulatory citation, indicating sampling options or modifications to be taken	Y	B2, 19-21	
b. Indicates how each sample/matrix type should be collected	Y	B2, 19-21, Appendix III and Appendix IV	

c. If in situ monitoring, indicates how instruments should be deployed and operated to avoid contamination and ensure maintenance of proper data	~~N~~ Y		The QAPP needs to clearly state that no in situ data are collected. The QAPP needs to clearly state that no continuous monitoring data are collected. **Response (date):** pg 20 B2 1st paragraph last sent **Resolved (date): Resolved 4/28/2014**
d. If continuous monitoring, indicates averaging time and how instruments should store and maintain raw data, or data averages			
e. Indicates how samples are to be homogenized, composited, split, or filtered, if needed	Y	B2, 19-21 B3, 21, Appendix III, Appendix IV, and Table 3	
f. Indicates what sample containers and sample volumes should be used	Y	Table 3, 40 + Appendix II	
g. Identifies whether samples should be preserved and indicates methods that should be followed	Y	Table 3, 40, Appendix II	
h. Indicates whether sampling equipment and samplers should be cleaned and/or decontaminated, identifying how this should be done and by-products disposed of	~~N~~ Y	Appendix III Appendix IV	The QAPP needs to describe or reference procedures for decontaminating samplers and sampling equipment between samples and the proper disposable of decontamination products. **Response (date):**pg 20: B2 1st paragraph added sent 2 & 3 **Resolved (date):** Resolved 4/30/2014
i. Identifies any equipment and support facilities needed	~~N~~ Y	B2, B3, B4 19-21	The field data collection forms in Appendices III and IV indicate additional support equipment not listed in Sections B2, B3 or B4. Please include a list of all support equipment needed for field sampling and measurements. **Response (date):** pg 20, B2 1st paragraph 4th sent added reference and App VIII Resolved (date): Please indicate where in the manual to find these instructions. Response (date): Resolved (date): Resolved 4/30/2014

j. Addresses actions to be taken when problems occur, identifying individual(s) responsible for corrective action and how this should be documented	Y	A4, 5; A7, 11 C1, 29	
B3. Sample Handling and Custody			
a. States maximum holding times allowed from sample collection to extraction and/or analysis for each sample type and, for in-situ or continuous monitoring, the maximum time before retrieval of information	Y	Table 3, 40	
b. Identifies how samples or information should be physically handled, transported, and then received and held in the laboratory or office (including temperature upon receipt)	Y	B3, 21; A9, 15-16; Appendix III and Appendix IV	
c. Indicates how sample or information handling and custody information should be documented, such as in field notebooks and forms, identifying individual responsible	~~N~~ Y	A9, 16, B3, page 21, Appendix III, Appendix IV, and Appendix V	For reference, please add where the EPA Region 8 Chain of Custody form is found in the QAPP (Appendix V) to the last sentence of the second paragraph in B3 on page 21 in the QAPP. **Response (date):**pg 22: B3 last paragraph, last sent added **Resolved (date): Resolved 4/28/2014**
d. Discusses system for identifying samples, for example, numbering system, sample tags and labels, and attaches forms to the plan	Y	Table 1, 37, Table 2, 38; B1, 17; B3, 21	
e. Identifies chain-of-custody procedures and includes form to track custody	Y	A9, 16; B3, 21, Appendix V	
B4. Analytical Methods			
a. Identifies all analytical SOPs (field, laboratory and/or office) that should be followed by number, date, and regulatory citation, indicating options or modifications to be taken, such as sub-sampling and extraction procedures	Y	B4, 21; Appendix I	
b. Identifies equipment or instrumentation needed	Y	Appendix I	

c. Specifies any specific method performance criteria	Y	Appendix I	
d. Identifies procedures to follow when failures occur, identifying individual responsible for corrective action and appropriate documentation	Y	B4, 21	
e. Identifies sample disposal procedures	Y	App. VI, Appendix I	
f. Specifies laboratory turnaround times needed	Y	B4, 21	
g. Provides method validation information and SOPs for nonstandard methods		N/A	Approved methods are used.
B5. Quality Control			
a. For each type of sampling, analysis, or measurement technique, identifies QC activities which should be **used, for example, blanks, spikes, duplicates, etc., and at** what frequency	Y	B5, 22-27	
b. Details what should be done when control limits are exceeded, and how effectiveness of control actions will be determined and documented	~~N~~ Y	B5, 23-24 D3, 32	Please describe in the QAPP the use of cation-ion balance information for understanding how surface water and groundwater may be impacted when sample error is ruled out as a factor. **Response (date):pg23: last paragraph** **Resolved (date): Resolved 4/28/2014** Please explain why 10% has been selected as criteria for the cation-anion balance evaluation. Most balances are 5% and approach a minimum of about 2%. **Response (date): revised to 5%, pg 23** **Resolved (date): Resolved 4/28/2014**

c. Identifies procedures and formulas for calculating applicable QC statistics, for example, for precision, bias, outliers and missing data	~~N~~ ~~Partially resolved-see additional comments in right column.~~ Y	B5, 24-27	Please include the minimum cation and anion tested species required for a cation-anion balance calculation. Please include equations and reference constants (citing sources) or reference for calculating cation-anion balances in the QAPP. **Response (date):**pg 24: create excel spreadsheet added; added language on pg 23 last paragraph **Resolved (date): partially resolved** Nitrate is frequently a significant contributor to the anions. Explain why this is not included or we suggest inclusion, since it is part of the testing suite. Response: Resolved: Resolved 4/30/2014 At the end of the year, when the QAPP is reviewed/revised for the next year, include the spreadsheet as used and with formula's shown in the revised QAPP. Add sentence on page 24 that indicates the spreadsheet and documentation will be added in the next revision of the QAPP. Response: Resolved: Resolved 4/30/2014 Please clarify the procedure for preparing samples for matrix spikes for trace elements in the Matrix Spikes subsection on page 27. The QAPP states the USGS NWQL will be doing this procedure. **Response (date):**corrected **Resolved (date): Resolved 4/28/2014** Use the Region 8 Laboratory acceptance criteria for evaluation the matrix spike data, which is included in Table 3. **Response (date):**corrected pg 27 last sentence **Resolved (date): Resolved 4/28/2014**
B6. Instrument/Equipment Testing, Inspection, and Maintenance			
a. Identifies field and laboratory equipment needing periodic maintenance, and the schedule for this	~~N~~ Y	B6, 27, Appendix I	Please correct citation in the last sentence of Section B6, Field Instrumentation Calibration on page 27 to the correct reference in the References section [Wilde, F.D, ed., variously dated, Field measurements:…] which includes sec. 6.0-6.8 and covers the instrument calibration of all field measurements for this project. **Response (date):**pg 27 revised under Field Calibration-6th sentence
b. Identifies testing criteria			
G c. Notes availability and location of spare parts			
d. Indicates procedures in place for inspecting equipment before usage			

e. Identifies individual(s) responsible for testing, inspection and maintenance			**Resolved (date): Resolved 4/28/2014**
f. Indicates how deficiencies found should be resolved, re-inspections performed, and effectiveness of corrective action determined and documented			
B7. Instrument/Equipment Calibration and Frequency			
a. Identifies equipment, tools, and instruments that should be calibrated and the frequency for this calibration	~~N~~ Y	B7, 27, Appendix I	See comment B6. **Response (date):** **Resolved (date): Resolved 4/28/2014**
b. Describes how calibrations should be performed and documented, indicating test criteria and standards or certified equipment			
c. Identifies how deficiencies should be resolved and documented			
B8. Inspection/Acceptance for Supplies and Consumables			
a. Identifies critical supplies and consumables for field and laboratory, noting supply source, acceptance criteria, and procedures for tracking, storing and retrieving these materials	~~N~~ Y	B8, 28	The QAPP needs to state that the Region 8 Laboratory will supply containers and preservatives for the project and reference the Lab QA Plan for description for the adequacy for use. **Response (date):pg 28: B8 1st sentence** **Resolved (date):** Please clarify if the tracking of containers, preservatives and other critical supplies will be documented on the field forms in Appendices III and IV. **Response (date):see above** **Resolved (date): Resolved 4/28/2014**
b. Identifies the individual(s) responsible for this	Y	B8, 28	
B9. Use of Existing Data (Non-direct Measurements)			

a. Identifies data sources, for example, computer databases or literature files, or models that should be accessed and used	~~N~~ ~~Partially resolved~~ Y	B9, 28	Please include discussion that the associated metadata for GIS-derived latitude and longitude coordinates are also collected in conformance with Federal and EPA Geospatial policy. See here: http://www.epa.gov/geospatial/docs/2131.pdf **Response (date):** **Resolved (date): Not Resolved** Please include sources and types of information the project requires to determine targeted sampling locations based on history of spills. Information must include locations, dates of spills, quantities spilled and hazards/chemical characteristics of spills. **Response (date):included weblink B9 last sentence** **Resolved (date): Resolved 4/28/2014** Please identify sources that are used for weather and climate data. **Response (date): ?** **Resolved (date): Resolved 4/28/2014** Existing data from NDDH and USACE need to be described here, including quality assurance review for acceptance for this project. Response (date): Resolved (date): Resolved 4/30/2014
b. Describes the intended use of this information and the rationale for their selection, i.e., its relevance to project	Y	B9, 28	
c. Indicates the acceptance criteria for these data sources and/or models	Y	B9, 28	
d. Identifies key resources/support facilities needed	Y	B9, 28	
e. Describes how limits to validity and operating conditions should be determined, for example, internal checks of the program and Beta testing	Y	B9, 28	
B10. Data Management			
a. Describes data management scheme from field to final use and storage	Y	B10, 28-29	
b. Discusses standard record-keeping and tracking practices, and the document control system or cites other written documentation such as SOPs			
c. Identifies data handling equipment/procedures that should be used to process, compile, analyze, and transmit data reliably and accurately	~~N~~ Y		Please correct the QAPP to state AWQMS (not STORET) is the database used by the Tribe to store and report data. **Response (date):Revised throughout the document** **Resolved (date): Resolved 4/28/2014**

d. Identifies individual(s) responsible for this	~~N~~ Y	B10, 28-29	The data management matrix on page 29 of the QAPP needs to state the Primary Responsibility is TAT Project Manager, and if appropriate include USGS for each item listed. **Response (date):**Revised pg 29 **Resolved (date): Resolved 4/28/2014**
e. Describes the process for data archival and retrieval	~~N~~ Y	B10, 28-29	Please state in the QAPP when or what frequency backups will be made of electronic files and where the backups will be located/stored. **Response (date):**pg 29: B10 1st paragraph **Resolved (date): Not Resolved** Please provide citation to support the "TAT Project Manager will utilize accepted data management procedures", or if already stated within the paragraph, strike this sentence. Response (date): Resolved (date): Resolved 4/30/2014
f. Describes procedures to demonstrate acceptability of hardware and software configurations	Y	B10, 28-29	
g. Attaches checklists and forms that should be used		N/A	
C. Assessment and Oversight			
C1. Assessments and Response Actions			
a. Lists the number, frequency, and type of assessment activities that should be conducted, with the approximate dates	~~N~~ Y	C1, 29-30	This multi-year QAPP needs to include a statement of an annual review and update for changes made during the year and for the new work plan using the Region 8 QA Document Crosswalk link: http://www2.epa.gov/sites/production/files/region8/qa/EPA_R8_QA_DocumentReviewCrosswalk.docx. **Response (date):**pg 31 C1 last paragraph last sentence **Resolved (date): Resolved 4/28/2014**
b. Identifies individual(s) responsible for conducting assessments, indicating their authority to issue stop work orders, and any other possible participants in the assessment process	Y	C1, 29-30	
c. Describes how and to whom assessment information should be reported	Y	C1, 29-30	
d. Identifies how corrective actions should be addressed and by whom, and how they should be verified and documented	Y	C1, 29-30	
C2. Reports to Management			

a. Identifies what project QA status reports are needed and how frequently	~~N~~ Y	C2, 31	The QAPP needs to state that annual review and revision of the QAPP and completed crosswalk will be submitted to the EPA Project Officer for EPA approval prior to the start of data collection for the upcoming year. **Response (date):**pg 31 C2 last sentence **Resolved (date): Resolved 4/28/2014**
b. Identifies who should write these reports and who should receive this information	Y	C2, 31	
D. Data Validation and Usability			
D1. Data Review, Verification, and Validation			
Describes criteria that should be used for accepting, rejecting, or qualifying project data	~~N~~ Y	D1, 31	Please correct the sentence to say "Data quality will be assessed by comparing entered data to original data **AND** by comparing results with the measurement performance criteria described…" Note that both of these processes comprise verification and validation. **Response (date):** **Resolved (date): Resolved 4/28/2014**
D2. Verification and Validation Methods			
a. Describes process for data verification and validation, providing SOPs and indicating what data validation software should be used, if any	~~N~~ Y	D2, 31	The statement that "analytical validation and verification methods are outside of the scope of the QAPP" needs to be removed. The QAPP needs to describe all project-related data verification and validation steps. Please note that the laboratory data review cannot satisfy the QAPP requirement for project-level review. **Response (date):**pg31: D1 removed language **Resolved (date): Resolved 4/28/2014** Please revise the sentences: "This information will be checked by the EPA Region 8 Laboratory to ensure that holding times have not been exceeded. Violations of holding times will be reported by the laboratory to the Environmental Director," to state that Region 8 laboratory reports results according to the Region 8 QA Plan section 4.10 to the client (TAT). **Response (date):**pg31-32: D2 revised language **Resolved (date): Resolved 4/28/2014** The QAPP needs to state that the Tribe in cooperation with USGS provides a project-level verification and validation of all data based on the project data quality objectives to determine usability of data, including qualifying data, as needed. **Response (date):** **Resolved (date): Resolved 4/28/2014**

b. Identifies who is responsible for verifying and validating different components of the project data/information, for example, chain-of-custody forms, receipt logs, calibration information, etc.	Y	D2, 31	
c. Identifies issue resolution process, and method and individual responsible for conveying these results to data users	Y	D2, 31	
d. Attaches checklists, forms, and calculations		N/A	
D3. Reconciliation with User Requirements			
a. Describes procedures to evaluate the uncertainty of the validated data	~~N~~ Y	D3, 32	The last sentence in Section D3 needs to state that **at a minimum** data verification and validation includes analyzing source solution blanks, equipment blanks, trip blanks, and reviewing EPA Laboratory QC data. **Response (date):**revised language **Resolved (date): Resolved 4/28/2014**
b. Describes how limitations on data use should be reported to the data users	~~N~~ Y	D3, 32	Please include, for reference, the description of qualifiers that could potentially be needed for each sample type and analytical method used for collecting data the project. Note that USGS has compiled a list with descriptions of common qualifiers. **Response (date):added Appendix IX, added reference language pg 14 and A7 Data Quality Objectives** **Resolved (date): Not Resolved. Appendix IX contains a list of various project data qualifiers used for other media programs from other sources. Appendix IX needs to be clear to only include the appropriate qualifiers used for this project or any water quality monitoring project.** **Response (date):** **Resolved (date): Resolved 4/30/2014**

Huff, Blake

From:	Pritt, Jeff
Sent:	Wednesday, April 30, 2014 2:31 PM
To:	Huff, Blake
Cc:	dprice@mhanation.com; Pfeiffer, Tricia; Berig, Jennifer; Brooks, Tom; Hanley, lee; Monson, Bill; Himmelbauer, Linda
Subject:	QA Final Review of TAT 106 2014 QAPP 4/30/2014
Attachments:	QA Review TAT 106 2014 QAPP_04-30-2014.docx; TAT_QAPP_04_30_14_FINAL DRAFT_4-30-2014 (2).docx

Hi Blake-

By conference call this morning with Dana Price, Tony Ranalli, Tricia Pfeiffer, and myself, the few remaining concerns were addressed jointly with the Tribe and USGS. Attached is the final draft of the QAPP. A final check of the web links in the QAPP resulted in the NOAA web link on page 28 not working.

The QA Program has completed its review of the Draft QAPP titled **Ambient Monitoring of Surface Water and Groundwater Quality on the Fort Berthold Indian Reservation, North Dakota.**

With the web link corrected, the QAPP is approvable. Please route it for signature.

Please route the QAPP for approval in the following manner:

- Obtain approvals on the final revised complete QAPP (including all Figures, Tables, SOPs, Attachments and Appendices).
- Route the complete QAPP for EPA approval. (The Regional QA Manager is the final signature.)
- Use the EPA routing slip (Web Forms version, OF41) to document EPA concurrence/approval routing of the final revised QAPP. Identify the document title on the Routing Slip and include all Region 8 individuals having concurrence and/or signature of the document. The routing slip is part of the official record (showing all individuals reviewing/concurring for approval).
- The QA reviewer (Jeff Pritt in this case) will attach the completed Crosswalk prior to the RQAM's signature, so he should be included on the routing slip prior to the RQAM.

The QAPP, QA Program QA Document Review Crosswalk (attached), and the Concurrence Routing Slip together constitute the QAPP document file. The QAPP document file, including the QAPP with original signatures, will be returned to you.

Please let me know if you have questions.

Jeff Pritt
U.S. EPA Region 8
1595 Wynkoop St, Denver, CO 80202
Ph: 303-312-7077
Fax: 303-312-6685