Underground Storage Tank and Petroleum Remediation Quality Assurance Program Plan

February 2018

Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, Minnesota 55155-4194

MINNESOTA POLLUTION CONTROL AGENCY

p-eao2-04

Section A.1: Approvals

Approval: **Date:**

Ronza B Jordan 4/3/18

Ronza Jordan, Acting Quality Assurance Manager
U.S. Environmental Protection Agency
Region 5
Land and Chemicals Division
(312)353-0849
jordan.ronza@epa.gov

Gary Victorine 4/6/2018

Gary Victorine, Chief
U.S. Environmental Protection Agency
Region 5
RCRA Branch
(312)886-1479
victorine.gary@epa.gov

Section A.1: Approvals (Continued)

Approval: **Date:**

for: **Nathan Blasing** — 3/20/18
Minnesota Pollution Control Agency
Supervisor, Tanks Compliance Unit
(218)316-3899
nathan.blasing@state.mn.us

Chris McLain — 3/20/18
Minnesota Pollution Control Agency
Supervisor, Petroleum Remediation Unit 2
Petroleum Remediation Program Manager
(651)297-8583
chris.mclain@state.mn.us

Michael Kanner — 3/20/18
Minnesota Pollution Control Agency
Manager, Petroleum and Remediation Section
(651)297-8505
michael.kanner@state.mn.us

Sarah Kilgriff — 3/20/18
Minnesota Pollution Control Agency
Manager, Land and Air Compliance Section
UST Program Manager
(651)757-2492
sarah.kilgriff@state.mn.us

Section A.1: Approvals (Continued)

Approval: **Date:**

Sarah Larsen 3/20/2018

Sarah Larsen,
Minnesota Pollution Control Agency,
Petroleum Remediation Program
Petroleum Remediation QA/QC Coordinator
(651)757-2517
sarah.larsen@state.mn.us

William Scruton 3/20/2018

William Scruton
Minnesota Pollution Control Agency
QA Coordinator
(651)757-2710
bill.scruton@state.mn.us

Kathryn Sather 3/20/2018

Kathryn Sather
Minnesota Pollution Control Agency
Division Director, Remediation Division
(651)757-2691
kathryn.sather@state.mn.us

Douglas Wetzstein 3/20/2018

Douglas Wetzstein
Minnesota Pollution Control Agency
Assistant Division Director, Industrial Division
(651)757-2819
doug.wetzstein@state.mn.us

Section A.2: Table of Contents

Section A.2: Table of Contents (Continued)

Section A.3: Distribution List

The listed individuals will receive copies of the approved QAPP and subsequent revisions, if applicable:

Ronza Jordan, *U.S. Environmental Protection Agency, (312)353-0849*
Gary Victorine, *U.S. Environmental Protection Agency, (312)886-1479*
Sherry Kamke, *U.S. Environmental Protection Agency, (312)353-5794*
Sarah Larsen, *MPCA, (651)757-2517*
William Scruton, *MPCA, (651)757-2710*
Luke Charpentier, *MPCA, (651)757-2268*
Michael Kanner, *MPCA, (651)757-2483*
Kathryn Sather, *MPCA, (651)757-2691*
Douglas Wetzstein, *MPCA, (651)757-2819*
Nathan Blasing, *MPCA, (218)316-3899*
Sarah Kilgriff, *MPCA, (651)757-2492*
Chris McLain, *MPCA, (651)757-2652*

Section A.4: Program Organization and Responsibility

The MPCA conducts program management for the Petroleum Remediation and Underground Storage Tanks Programs. The Petroleum Remediation Program is responsible for the protection of human health and the environment by evaluating, minimizing, or correcting petroleum contamination impacts to soil and water caused by leaking storage tank systems. The Underground Storage Tank Program was created to help prevent contamination caused by leaking tanks. This program focuses on technical assistance, education and training, inspection, enforcement and certifying contracts, and installing, removing, and repairing tanks to achieve this objective. The organizational structure of the MPCA is shown in Figure 2 in the Minnesota Pollution Control Agency's Quality Management Plan (January, 2013).

Section A.4.1: The MPCA Program Managers

The MPCA Petroleum Remediation/UST Program Managers will:

- Provide administrative direction to assigned staff and to the MPCA QA/QC coordinator as needed.
- Implement the elements of the program as well as any required quality control measures.
- Manage the budget to assure that goals are met and funds and resources are responsibly allocated.
- Oversee the preparation of an annual program summary to include measurable benchmarks, problems encountered regarding QA/QC, and recommended changes in procedures.
- Review all project deliverables and strategies.
- Provide direct supervision and project assignment to assigned staff.
- Serve as primary contact with the US EPA.
- Provide technical direction for the preparation of work plans and the tasks to be performed.
- Conduct annual performance appraisals of assigned staff specific to their position description relating to the Petroleum Remediation/UST Programs.
- Review and approve the QAPP including subsequent revisions.

Section A.4.2: MPCA Petroleum Remediation QA/QC Coordinator and the MPCA QA Coordinator

The MPCA QA Coordinators will:

- Represent the MPCA with the contractor(s) ensuring adequate exchange of information regarding program responsibilities and effective functioning of the analytical program.
- Coordinate analytical needs and projections, analytical data reports from the contractor, and resolution of problems arising from contract provisions with the analytical laboratories and MPCA staff.
- Notify the contractor of updates and changes in analytical techniques or requirements of federal and state regulatory programs.
- Review invoices to ensure proper billing for services provided by the contractor(s).
- Update and distribute the Petroleum Remediation Program/UST QAPP when deemed necessary with rule, statute, policy, procedure, technology changes, or when US EPA approval expires.
- Provide an overview to the Program Manager of analytical results and quality control data to ensure the laboratory has met program requirements.

Section A.4.3: MPCA Petroleum Remediation/UST Supervisors

The MPCA Petroleum Remediation/UST Supervisors will:

- Provide direct supervision and project assignment to assigned staff.
- Provide direction for the daily work activities.
- Provide technical representation at meetings.
- Provide direction for sampling requirements.
- Prepare reports.
- Review and approve the QAPP including subsequent revisions.

Section A.4.4: MPCA Remediation/Industrial Division Directors

The MPCA Remediation/Industrial Division Directors will:

- Provide oversight for the implementation of the QAPP.
- Provide administrative direction and direct supervision of staff.
- Provide management representation at meetings.
- Manage the budget to assure that goals are met and funds and resources are responsibly allocated.
- Review and approve the QAPP including subsequent revisions.

Section A.5: Definition/Background

There are over 18,000 above-ground and underground storage tanks (UST) in use in Minnesota. A large number of these tanks are regulated by state statutes and rules. The Storage Tank Program provides technical assistance to owners/operators about tank requirements and spill prevention. This assistance includes tank inspections, numerous fact sheets, and an Agency Web page (https://www.pca.state.mn.us/waste/storage-tanks). The Program also evaluates compliance of the tank facilities with the regulations through inspections, investigations, and enforcement actions. Furthermore, the Program ensures the proper installation, removal, and repair of tank facilities by certifying contractors conducting that work.

The purpose of the Petroleum Remediation Program is to protect human health and the environment by evaluating, minimizing, or correcting petroleum contamination impacts to soil, water, and air caused by leaking storage tank systems. Regulatory authority for the program is given in Minnesota Statute Chapter 115C, "Petroleum Tank Release Cleanup Act." The Program oversees the prompt investigation, cleanup, and closure of petroleum tank release sites. Program requirements and technical guidance are provided through fact sheets available on the Agency Web page (http://www.pca.state.mn.us/waste/petroleum-remediation-program).

Section A.6: Program Descriptions

Section A.6.1: Objective

The objective of the MPCA's UST/Petroleum Remediation Programs is to evaluate and remove risks to human health and the environment from petroleum product releases (either by spills or leaking UST). Strategies include (1) compliance assistance to promote understanding of environmental regulations; (2) offer incentives that encourage facilities and responsible parties to identify and cleanup releases; (3) educating, training, and certifying tank contractors; (4) monitor compliance through inspections and investigations; and (5) conduct civil and criminal enforcement actions to correct violations and deter future noncompliance. The MPCA will strive for the performance objectives set forth in the Environmental Performance Partnership Agreement (EnPPA) between the U.S. Environmental Protection Agency (EPA) Region 5 and the MPCA (See Section D.3: Reference 6 for EnPPA Goals, Outcome Objectives, Key Measures, and Strategies). This QAPP falls under all requirements of the MPCA's QMP which is approved by EPA Region 5.

Section A.6.2: Scope

The scope of the UST/Petroleum Remediation Programs, including a description of MPCA goals, is defined in the EnPPA. As part of the overall Petroleum Remediation Program, soil, water and air samples may be collected and analyzed. To assure data quality, the EPA has required the MPCA to develop a Quality Assurance Program Plan (QAPP). The objective of the QAPP is to define the Quality Assurance and Quality Control (QA/QC) procedures to be followed for the collection and analysis of environmental samples to assure sufficient precision and accuracy of results in order for them to be used for their intended purpose. The quality objectives will generally follow the guidance outlined on the MPCA Quality System webpage: (http://www.pca.state.mn.us/about-mpca/mpca-quality-system). The Quality System for MPCA's environmental data describes the agency's general policy for data quality assurance.

Section A.6.3: Purpose/Background

Since 1988, the MPCA has been authorized under a cooperative agreement with the EPA, Region 5, to provide technical assistance and to conduct tank inspections in the State of Minnesota. State Program approval was given to the MPCA in 1999 to administer the tanks program in Minnesota and the MPCA is currently pursuing State Program approval again. Inspections are conducted to determine whether a tank facility complies with the federal and state regulations. The QAPP supports the scope of the UST/Petroleum Remediation Programs under the MPCA and EPA EnPPA.

Section A.6.4: UST Inspections

Subtitle I of the Solid Waste Disposal Act created the UST Program. The Energy Policy Act of 2005 (in Title XV, Subtitle B) contains new amendments for the UST Program. Sections of the Acts provide authority for conducting inspections to monitor compliance and provide guidelines which identify the USTs that are required to be inspected, what the requirements are for the inspection, who can perform the inspection, and what information needs to be reported to the regulatory authority.

Section A.6.5: Sampling at Petroleum Remediation Sites

Sampling is conducted during an investigation of a petroleum release site to identify the presence of any contamination related to a release of petroleum product or to confirm information provided by the facility. Sampling techniques include the collection of water samples, soil samples, or air samples, as appropriate. In all instances, analytical results of the sampling provide information to the regulatory authorities to establish the compliance of the facility with Petroleum Remediation guidance.

Section A.6.6: Analytical Samples

The samples are brought under chain of custody procedures to an approved laboratory (either the MDH Environmental Laboratory or a MDH-certified laboratory). The samples are labeled to allow identification of each sample specific to where on site the sample was taken. The sampler identifies the type of sample and the action level upon the chain of custody. This information allows the laboratory to use proper methods that bracket the action level when analyzing these samples. Specific instructions on sample collection and target analytes of concern are provided in http://www.pca.state.mn.us/publications/c-prp4-04.pdf, http://www.pca.state.mn.us/publications/c-prp4-05.pdf, http://www.pca.state.mn.us/publications/c-prp4-01a.pdf, and. http://www.pca.state.mn.us/publications/c-prp4-21.pdf.

Section A.6.7: Intended Data Usage

The data is used to determine:

* Whether a release has in fact occurred at a site;
* The extent and magnitude of contamination at a site;
* The quality of data generated by responsible parties (through the use of split sampling);
* Appropriate disposal of contaminated soils during site remediation activities; and
* The potential and/or immediate risk to human health and the environment associated with a site or facility.

Section A.6.8: Annual Technical Reports

The MPCA prepares an annual report at the end of each federal fiscal year. The annual report will include a progress report on the program goals established in the EnPPA. The annual report will be distributed to the appropriate MPCA staff and to the U.S. EPA Region 5 Project Manager.

Section A.7: Quality Assurance Objectives and Criteria

Section A.7.1: Overview

The Data Quality Objectives (DQOs) for the Petroleum Remediation Program are determined by the site requirements. For example, the required limits of a matrix (for example water, see Section A.6.4.4) and site requirements (such as residential) will determine the reporting limit needed from the laboratory. This directly influences Step 3 of the DQO process by determining the limit needed. The MPCA technical staff determine the required number of samples, the reporting limits, the level of quality from the laboratory (rush vs. standard vs. legal), the sampling locations, and the reporting requirements.

Quality assurance objectives are developed for field sampling, chain of custody, laboratory analysis and reporting (see detailed procedures in Section B.2 and B.3). Meeting these objectives will provide the MPCA with defensible information to be used:

* To identify pollutant sources;
* To determine the quality of data generated by responsible parties through the use of split samples;
* For enforcement actions; and
* For litigation if necessary.

The responsible party is responsible for field sampling and chain of custody until the laboratory accepts samples. Specific procedures to be used for sampling, quality control, audits, preventive maintenance and corrective actions are described in other sections of this document. The purpose of this section is to define quality assurance goals for precision, accuracy and completeness. Establishing these goals allows the State to judge the adequacy of the results and whether corrective actions are necessary.

The quality assurance objectives to be met for both field operations and laboratory activities are discussed below.

Laboratory reports include the date of sampling, the date of analysis, the signed Chain of Custody form, a narrative of the analysis which notes items that are outside the laboratory QC limits, and the analytical results for the collected sample. In addition to the analytical results, the reports include the percent recoveries (% R) of surrogates and the percent recoveries (% R) and relative per cent differences (RPD) of laboratory control sample/laboratory control sample duplicates and matrix spike/matrix spike duplicates.

Section A.7.2: Blanks

A trip blank is typically utilized with the collection of samples to be analyzed for volatile hydrocarbons to determine whether contaminants are introduced into samples due to improper handling techniques or contaminants have permeated the cap of the sample vial during shipment. Volatile samples will be collected for this program and therefore trip blanks will be utilized for quality assurance purposes.

The samplers use field blanks as equipment blanks while sampling. The sampler takes de-ionized water and pours it through any device used in sampling (such as a bailer or sampling scoop) to ensure that there are no background levels of contaminates. Field blanks are submitted at a 5% rate to the laboratory. Field blanks results verify that the field sampling and laboratory procedures are free of contamination and do not contaminate blank samples.

The laboratory uses method blanks to verify the extraction procedures, glassware, and instrument conditions have background below the laboratory reporting limits. The method blanks are reported with MPCA samples to allow the project manager to determine that laboratory contamination or analytical error could cause a false positive. The laboratory performs method blanks at a rate of one for each analytical batch of twenty samples (5%) or less to ensure a contaminant-free environment.

Section A.7.3: Duplicate Samples

As is the case for field blanks, duplicate samples are collected as necessary to protect the integrity of the sampling investigation. Duplicates are collected by alternately filling two separate sample containers from the same source for each set of parameters. Duplicate sample analyses provide a check on sampling and analytical reproducibility, or precision. For soil matrices, one duplicate is generated for each ten samples collected. The laboratory also prepares and analyzes matrix spike and matrix spike duplicates (MS/MSD) to gain a measure of reproducibility. MPCA has a relative percent difference (RPD) goal for duplicates of 25% for water and 50% for soils.

Section A.7.4: Spike Samples

Spiked samples will not be collected in the field but MPCA does submit adequate volumes of samples to ensure the laboratory has enough sample to allow for spike and spike duplicate analyses. MPCA policy allows a maximum recovery of 150% and a minimum recovery of 30%. The laboratory uses MS/MSD recoveries to measure accuracy in the analyses. Laboratory-generated limits for spike recoveries are used in validation of data (when required). Staff sampling for analytes of concern indicate on the COC which samples are collected for spike and spike duplicate samples. MPCA policy requires a 10% rate of spikes for environmental samples.

Section A.7.5: Laboratory Activities

The quality assurance objectives for accuracy, precision, completeness, representativeness, reporting limits, and comparability to be met by the laboratory are described in the laboratory's Quality Assurance Manual (QAM). Every laboratory that works on a Petroleum Remediation project must submit their QAM to the MPCA.

Section A.7.6: Definitions of Precision, Accuracy, Representativeness, Comparability, and Completeness

Section A.7.6.1: Precision

Where possible, laboratory precision is measured through the collection and analysis of duplicate samples. Duplicates are collected at the rate of one duplicate per ten environmental samples collected. The result for the duplicate sample is compared to the result of the known sample. The relative percent difference (RPD) between the known sample result and the duplicate sample result is calculated according to the following formula:

$$RPD = \frac{(Sample\ Conc. - Duplicate\ Conc.)*200}{(Sample\ Conc. + Duplicate\ Conc.)}$$

Precision can also be determined between the results of a matrix spike (MS)/matrix spike duplicate (MSD) pair or between a laboratory control sample (LCS)/laboratory control sample duplicate (LCSD) pair. RPD results should be < 25% for water samples and < 50% for soils samples for the data to be acceptable.

Section A.7.6.2: Accuracy

The accuracy of the measurement is gauged through the analyses of surrogate spikes, matrix spike (MS)/matrix spike duplicate (MSD), and/or laboratory control sample (LCS)/laboratory control sample duplicate (LCSD). Surrogate compounds are spiked into every sample prior to extraction and analysis. Where possible, MS and MSD samples are collected at the rate of one set per 20 environmental samples. If an MS/MSD pair cannot be analyzed, an LCS/LCSD pair may be used to measure accuracy. The percent recovery is determined by comparing the spiked sample concentration to the environmental (unspiked) sample concentration. The formula for determining percent recovery is as follows:

$$\%R = \frac{(Spiked\ Sample\ Conc. - Environmental\ Sample\ Conc.)*100}{(Spiked\ Concentration\ Added)}$$

Acceptable data falls between 30% and 150% recovery.

Section A.7.6.3: Representativeness

Representativeness of the data set is the measure that expresses the degree to which the data accurately represents the population as a whole. The methods for sample collection in the field, sample preservation, transportation to the laboratory, sample preparation, and sample analysis are reviewed to determine if appropriate procedures were followed. If the procedures as described in this QAPP were followed, sample results are considered representative of the site.

Section A.7.6.4: Comparability

Comparability is the degree of confidence that one data set can be compared to another data set and whether the data sets can be combined and used for decision-making purposes. The level of comparability between data sets is determined by reviewing sample collection and handling procedures, sample preparation and analytical procedures, holding times, and quality assurance protocols. When a large difference in one of the methods or procedures exists, the comparability of the data is considered low. If all of the procedures were followed, data from the same site is considered comparable.

Section A.7.6.5: Completeness

Completeness is measured by determining the ratio of valid sample results compared to the total number of samples for a specific matrix. During data verification, the data completeness is determined by the following equation:

$$\% \text{ Complete} = \frac{(\text{\# of Valid Results}) * 100}{(\text{\# of Samples Tested})}$$

A completeness of 90% or better must be obtained in order for a laboratory report to be considered acceptable. Laboratory reports which are not at least 90% complete are rejected. If the laboratory is at fault, they will be responsible for securing the re-collection and re-analysis of samples.

Section A.8: Specialized Training/Certifications

Section A.8.1: Field

Field personnel who will be working with potentially hazardous substances have 40-hour OSHA training and yearly 8-hour refresher training. Copies of this training are maintained by the consultants and are made available to the MPCA upon request. Inspectors have also participated in training provided by the EPA for conducting UST inspections. Upon EPA approval, the inspectors receive the credential for conducting UST inspections. The field samplers are also trained in specific sample collection and chain of custody procedures. Copies of SOPs are also sent out in the field with the samplers.

Section A.8.2: Laboratory

Laboratory personnel have been trained in proper analytical techniques. They also receive annual refresher training on such items as laboratory safety, right to know, and emergency procedures. The documentation of this training is maintained in the laboratory's QA Office.

Certification of environmental laboratories is provided by the Minnesota Department of Health. The laboratory must maintain certification for Petroleum Remediation analyses in water, solid and chemical materials, and air during the length of the project.

Section A.9: Record Keeping

The MPCA retains sampling records from UST/Petroleum Remediation inspections and investigations within the files constructed and maintained specific to each case. Sampling records include all information related to the sampling event, including date, site location, maps or diagrams of sample sites, chain of custody paperwork, and analytical results. Files for each UST inspection or PRP investigation are kept for five years in active files, and then are archived. In both instances, the files are retrievable.

The laboratory SOP for records retention indicates that all data documentation, records, protocols, and final reports are stored either on-site at the laboratory or off-site in secure storage. The records are retained for a period of not less than five years.

Section B: Data Generation and Acquisition

Section B.1: Sampling Design

The UST Program does not collect samples. Tank owners/operators hire contractors for this work. In the Petroleum Remediation Program, the majority of the sampling is also contracted by the responsible party. Staff may collect a limited number of samples for QC purposes. The data derived from the analysis of samples acquired during site investigations is used as the basis for appropriate remedial action and subsequent enforcement actions. The objective of sampling is to obtain data, which will assist MPCA investigative personnel in the identification and confirmation of the release of petroleum products into the environment. This information is also used in enforcement proceedings that may result from a facilities noncompliance with UST/Petroleum Remediation regulations.

Section B.2: Sampling Procedures

Prior to sample collection, field personnel coordinate with the laboratory to assure that appropriate equipment and supplies are available to meet the sampling need. Sampling techniques employed by the MPCA are appropriate for water, soil, and air samples.

The field sampling procedures and required analyses are outlined on the MPCA PRP Website (see Section A.6.6). More detailed information should be defined in an approved site-specific QAPP or Sampling and Analytical Plan (SAP). After collection, the samples will be properly preserved and shipped to the laboratory for analysis.

Section B.3: Sample Custody

Section B.3.1: Overview

Due to the evidentiary nature of samples collected during enforcement investigations, sample possession must be traceable from the time samples are collected until they are disposed of. To maintain and document sample possession, chain of custody (COC) procedures are followed.

Section B.3.2: Field Custody Procedures

Trained field personnel collect the samples (see Section B.1). The field personnel either have the samples in their possession, in their view, in a secured area that only they have access to, or turn custody over to another individual who has signed the chain of custody (COC) form (See Attachment 3 for an example COC form). The COC is the record of all individuals who come in contact with the samples. A copy of the chain of custody is maintained at all times to ensure the samples can be used in for enforcement. A COC has the following information present:

A. Date and time of sampling,
B. Name of sampler,
C. Identification number of the samples,
D. Analytical methods requested,
E. Information to the hazard of the sample,
F. Project name,
G. Signature of the sampler, and
H. MPCA contact name and phone number.

Sample custody is maintained from collection through analysis. The samples are cooled on ice. The chain of custody form is signed by the sampler and double zip-locked and taped to the inside lid of the cooler. The cooler is custody taped on two corners and shipped. The sampler and the laboratory keep a copy of the bill of lading as proof of custody in shipment. Records of custody are maintained by the MPCA within the site files.

Section B.3.3: Laboratory Custody

Laboratory custody procedures are usually described in the laboratory QAM. The laboratory signs the COC when the samples are received. The laboratory verifies the COC is correctly filled out and all samples are accounted for (and not broken). Any problems that occur upon receipt of the samples will cause the sample clerk at the laboratory to immediately contact the MPCA QA Coordinator. The MPCA will decide if the samples are to be run depending on the problem. The laboratory logs in the samples into the laboratory LIMS system. The system assigns a unique number to each sample. The log-in numbers are then used to track the sample at the laboratory.

The laboratory stores the samples in a secure refrigerated area that maintains the samples at 4° +/- 2° C. The sample holding area is secure from unauthorized personnel having access to the samples. The samples are removed by an analyst for extraction/digestion, the extraction/digestion performed, and any remaining sample placed back in the refrigerator. The laboratory disposes of the samples, except in case of very hazardous samples, which are returned to the site or lab-packed for disposal at an appropriate facility.

Section B.4: Analytical Methods

Specific instructions on analytical methods are provided in
http://www.pca.state.mn.us/publications/c-prp4-04.pdf,
http://www.pca.state.mn.us/publications/c-prp4-05.pdf,
http://www.pca.state.mn.us/publications/c-prp4-01a.pdf, and
http://www.pca.state.mn.us/publications/c-prp4-21.pdf.

The laboratory SOPs for the UST/Petroleum Remediation analyses must be supplied to the MPCA for review before project implementation.

Section B.5: Quality Control

Field and laboratory QC checks are identified in Table 1. The frequency of analysis and the control limits are also listed. If the results don't meet the QC acceptance criteria, corrective actions are defined.

Table 1: Quality Control Elements

QC Type	Soil	Water	Soil Vapor
Blanks			
Field Blanks		X	---
Method Blanks	X	X	---
Spikes			
Matrix Spike	X	X	---
Matrix Spike Duplicate	X	X	---
Laboratory Control Sample	X	X	X
Surrogates	X	X	---
Calibration Checks	X	X	X
Duplicates			
Field Duplicates	X	X	X
Laboratory Duplicates	X	X	X

Section B.5.1: QC Type

Section B.5.1.1: Field Blanks

Field blanks are collected to show any bias that is related to collection equipment or transport of samples from the field to the laboratory. One field blank is collected for each day's set of water or wipe samples. If there is contamination in the field blank but not in the samples, no action is required. Any positive UST/PRP result which is associated with a positive UST/PRP result detected in a field blank is evaluated. If the environmental sample result is less than X5 the concentration detected in the field blank, the report level is raised to the UST/PRP concentration found in the sample. If the environmental sample result is greater than X5 the concentration found in the field blank, no qualification is necessary. However, an explanation of the rationale should be provided in the narrative accompanying the report.

Section B.5.1.2: Method Blanks

One method blank is extracted and analyzed with each batch of up to 20 samples to demonstrate that there are no interferences from the glassware, reagents, and analytical system. UST/PRP chemicals of concern should not be present in the method blank at ½ the report level concentration. If any method blank shows target analytes above ½ the report level, a solvent blank should be injected to demonstrate that there was no carry-over from standards or samples. If there was carry-over, clean the analytical system and re-inject the method blank. If the method blank contamination cannot be attributed to carry-over, the samples that were associated with the blank should be re-extracted and re-analyzed.

Section B.5.1.3: Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

Matrix spike/matrix spike duplicate (MS/MSD) pairs are used to determine if there are any effects related to the sample matrix. One pair should be spiked, extracted, and analyzed per batch of up to 20 samples. The % recoveries of the MS/MSD pairs are used to measure accuracy of the analysis while the relative percent difference is used to measure precision. The % recoveries should be 30-150% and RPD should be ≤ 25% for water matrix while it should be ≤50% for all other matrices.

Section B.5.1.4: Laboratory Control Sample (LCS)

A laboratory control sample (LCS) is an aliquot of clean matrix and of the same weight or volume as the environmental samples. One LCS is prepared with each batch of up to 20 samples. The LCS is spiked with the same target analytes and at the same concentration as the MS. The % recoveries of the LCS are used to show that the analysis is in control if there is a matrix effect associated with the analysis of the sample matrix in the MS/MSD. The % recoveries should be 30-150% for all matrices.

Section B.5.1.5: Surrogate Analytes

Surrogate analytes are added to all blanks, samples, matrix spikes, and laboratory control samples to monitor method performance. The % recoveries of the surrogates should be 30-150% for all matrices.

Section B.5.1.6: Field or Laboratory Duplicates

Field and laboratory duplicates are used to measure precision. One pair should be extracted and analyzed per ten samples or less. The RPD should be ≤ 25% for a water matrix while it should be ≤50% for all other matrices.

Section B.5.1.7: Out-of-Control Situations

When the out-of-control situations listed in Sections B.5.1.3 through B.5.1.6 occur, the failing analysis should be re-injected into the analytical system. If the re-analysis meets QC criteria, report the second analysis. If the re-injection still does not meet criteria, the affected samples should be re-extracted and re-analyzed. If the results of the re-analysis of the MS/MSD pair still fail to meet criteria and the result of the LCS is acceptable, then the problem is related to matrix and the QC batch requirements are considered to have been met. Report the results of the batch and qualify the result of the environmental sample chosen for QC purposes as estimated. If the results for the LCS fail again, instrument maintenance is required. After the maintenance has been completed, another initial calibration must be performed.

Section B.6: Instrument/Equipment Testing, Inspection, and Maintenance

Section B.6.1: Field Equipment

Delays in project schedules, poor output in performance, and erroneous results in investigative operations can result from improperly maintained equipment. Therefore, preventative maintenance of field equipment is performed routinely before each sampling event. More extensive maintenance is performed based on hours of use and manufacturer recommendations. Spare parts for all field equipment as well as back up instruments are kept in the MPCA Field Operations Center (FOC). The FOC performs preventative maintenance on a routine schedule on all field equipment for the MPCA. Program inspectors and samplers are anticipated to need little field instruments. Standardized field sampling equipment (bailers, scoops, bowls, push probes, etc.) will be maintained by the FOC.

Section B.6.2: Laboratory Equipment

The protocols for testing, inspection, and maintenance of laboratory equipment are addressed in the laboratory QAMs. Additionally, the laboratory's standard operating procedures (SOPs) present the specific protocols to be followed as part of the analysis for UST/Petroleum Remediation. The preventative maintenance program employed by the laboratory is described in the laboratory QAM. In general, the preventative maintenance is performed on a scheduled basis on all instruments in the laboratory. The preventative maintenance performed is documented in the instrument maintenance logbooks kept at the instrument. Irregularities noted during operations are traced through the maintenance logbook to allow for efficient corrective action to solve problems. Analysts are trained in preventative maintenance of their assigned instruments. The laboratory utilizes in-house service technicians in the event of instrument failures. Contracts are maintained on the computer hardware and software. Backup instrumentation is generally available if a specific analytical system becomes unavailable.

Section B.7: Instrument/Equipment Calibration and Frequency

Section B.7.1: Overview

This section discusses calibration procedures for field and laboratory instruments to be used for the UST/PRP Compliance Program. All laboratory equipment used for analytical determinations is subject to periodic inspection and calibration. Frequency of calibration is based on the type of equipment, inherent stability, manufacturer recommendations, and intended use.

Section B.7.2: Field Procedures

UST Program staff do not use field equipment. If field equipment is used by the Petroleum Remediation Program staff, they follow the calibration procedures described in the manufacturing instructions and/or Program SOPs.

Section B.7.3 Laboratory Procedures

The calibration procedures followed by the laboratory are outlined in the Laboratory QAMs and SOPs. The basic procedure for the analyses is to calibrate the analytical instruments at five levels. One of the levels must be at or below the report level for the individual target analyte. The initial curve must have a coefficient of ≥0.99 or a %RSD of ≤20%. The five-point initial calibration curves are verified with an external source calibration standard and then routinely (as specified in the MDH Certification Rule or laboratory SOP) with a calibration verification check standard. All calibration standards must have a percent difference (%D) of <15%.

Section B.8: Inspection/Acceptance of Supplies and Consumables

An MPCA staff person inspects all supplies and consumables for integrity and suitability for use. Any supply or consumable judged to be of inferior quality or not suitable for the intended use is rejected. Sample containers are pre-certified as clean by the laboratory.

All chemicals and solvents used in the laboratory are inspected to verify that they are of the appropriate grade for their intended use. All consumables found to be contaminated are removed from use. The laboratory has a tracking system that incorporates the date of receipt, the date the container is opened, and the assigned expiration date of the chemical or standard. The procedures are documented in the individual laboratory Quality Assurance Manual.

Section B.9: Non-direct Measurements

Historical data may be used to initiate an investigation. However, all decisions as to whether a site is compliant with the policy outlined in federal or state rules are based on samples collected during an inspection.

Section B.10: Data Management

Section B.10.1: Data Recording

Data and information collected in the field will be recorded in dedicated notebooks and forms. Data recording procedures to be followed by the laboratory are discussed in individual laboratory Quality Assurance Manual.

Section B.10.2: Data transformation

Data and field information is transformed in various MPCA offices. Procedures for data transformation by the laboratory are discussed in the laboratory Quality Assurance Manual. Data are input into various computer programs for storage. The programs utilized include Microsoft Access®, Excel® and Word®.

Section B.10.3: Data Transmittal

Data and field information are delivered to the MPCA using raw data notebooks and forms. Analytical data are submitted to the MPCA as final analytical reports. These reports have been reviewed and approved by the laboratory's technical, QA/QC, and project management staff. Some data is now sent to the MPCA electronically. Lab_MN is an electronic data deliverable (EDD) format developed by the MPCA and MN.IT enabling labs to submit results in an EQuIS-ready format. It incorporates EQuIS data standards and enables remote error checking prior to submitting data to the MPCA. An annual report of Program activities is prepared at the end of each federal fiscal year.

Section B.10.4: Data Rejection

Analytical data which does not meet the established QA/QC criteria defined in this QAPP is rejected. Field data is evaluated by the technical staff to ensure that it is compliant with the QAPP. Data collected that is judged to be out of compliance are qualified, rejected, and re-collected if possible.

Section B.10.5: Data Tracking

MPCA staff contact the analytical laboratory on a regular basis regarding the status of sample analysis.

Section B.10.6: Data Storage and Retention

For MPCA, data storage and retention is dictated by Minnesota statute and department policy. Official laboratory records are managed using an inventory of records with a schedule establishing retention periods and disposal requirements.

Section C: Assessment and Oversight

Section C.1: Response Actions

Section C.1.1: PRP Site Decision Committee (SDC)

An Internal Assessment Review process is in place for the PRP that ensures management controls are in place and that they are carried out by the organization in order to plan, implement, and assess the results of the project.

Section C.1.2: PRP Field Audit Program

A thorough audit of the field sample collection activity is made. This audit reviews equipment, personnel, training, field documentation (photographs, daily field logs, and checklists), and chain-of-custody records to ensure compliance to the QAPP. The results of the audits (and any identified corrective actions) are summarized in a report to management.

Section C.1.3: Laboratory Audits

Section C.1.3.1: Internal Audits

The laboratory QA staff conducts internal audits of all departments involved with the handling/analysis of the Petroleum Remediation samples. These internal audits take place on an annual basis. These audits review the quality policies and implementation of the policies at the laboratory. The reports of these audits are sent to the laboratory manager and quality assurance officer for review and improvement in operations. The audit concentrates on the specific SOPs in each section, quality assurance practices, sample handling, documentation, and follow-up on prior audits. These audits are used by the laboratory to identify any problem in their operations before there is an effect to the data. All audits are documented and kept in the QA office. If problems occur or corrective action is initiated, the QAC from MPCA is contracted immediately for assistance in corrective actions. Copies of the internal audit findings (along with any required corrective actions) are submitted to the MPCA's QA Coordinator. As a result of the internal audits, the MPCA may audit at its discretion.

Section C.1.3.2: External Audits

External audits of the laboratory are performed by the Environmental Laboratory Section of the Minnesota Department of Health. Copies of the findings of these external audits (and any identified corrective action) are submitted to the MPCA's QA Coordinator. As a result of these external audits, the MPCA may audit at its discretion.

Section C.1.3.3: Performance Evaluation (PE) Studies

The laboratory analyzes Performance Evaluation Samples (PE Samples) which are blind samples prepared by external companies and shipped directly to the laboratory. The samples are logged in and analyzed as standard samples with the results being reported back to the independent company for scoring. The laboratory receives these scores and reports them to regulatory authorities (or states requiring PE samples for certification). Satisfactory performance must be maintained over the effective time of the QAPP. Copies of the results of the PE studies must be supplied to the MPCA's QA Coordinator.

Section C.2: Corrective Action/Reports to Management

For each analytical activity employed in this program, the laboratory regularly tracks the overall quality assurance issues. When a quality control sample or QA issue is found to be out of control, Corrective Actions (CA) are implemented. Corrective action includes re-analysis of samples, re-sampling, flagging of data, or rejection of the data. MPCA is informed of any major CA that is performed on any Program sample.

Section C.2.1: MPCA Corrective Actions

The individual identifying a potential issue first documents the problem in the field notebook. The project manager who has final sign-off authority on any problem or issue tracks the problem. The project manager tracks all CA. The PM is responsible for identifying the problem, verifying proper documentation is written and implementing the correct action. The project manager will place final documentation into the site record. Any major CA involving the laboratory is tracked by the both the laboratory QAO and the MPCA project manager. The MPCA project manager has final sign-off authority on issues dealing with Program samples.

Section C.2.2: Laboratory Corrective Actions

Laboratories have a corrective actions system that is described in the laboratory QAM. Generally, an individual involved in the analysis of the samples or review of the data discovers the problem. The problem is identified and documented. The documentation is important to allow tracking of the problem and ensure a proper solution is implemented. All analysts, QA staff, and managers/supervisors must agree to the solution to the problem. The QA staff will go back and verify that the solution corrected the problem. The documentation is archived with the client project folder.

Section C.2.3: Laboratory Reports

The laboratory sends a complete report to the MPCA that includes the following information:

a. A narrative discussing overall issues with the data (e.g. calibration, holding times, internal QC, etc.),
b. Extraction date,
c. Sampling date,
d. Analysis date,
e. Alphabetical list of compounds,
f. Reporting limits,
g. Method of analysis and extraction,
h. Signature of a laboratory officer,
i. Chain of custody,
j. Results of spike,
k. Spike duplicates,
l. Results of surrogate samples,
m. Blanks, and
n. Concentrations found of each analyte.

The laboratory report is given a final review by the laboratory project manager, then signed, and sent to the MPCA. Specific procedures used by the laboratory will be found in the QAM.

Section C.2.4: Reports to Management

An annual report summarizes the program's sampling and analytical activities for the previous year, the findings of the audits, any required corrective actions, the results of PE studies, any data quality problems (along with purposed solutions), any major changes in personnel, and an overall evaluation of the laboratory's quality assurance program. The report is sent to all individuals identified in Section A.4.

Section D: Data Validation and Usability

Section D.1: Data, Reduction, Verification, and Validation

Section D.1.1: Data Reduction

In general, instrument response for the quantitative analytical procedures described in the Petroleum Remediation SOPs, is converted to concentrations or absolute amounts of analyte by use of a multipoint calibration curve which relates instrument response to the quantity of the analyte introduced to the instrument. The analyst reduces the raw data produced by the instrument using equations found in the laboratory SOP or QAM. Technical expertise of the analyst is needed for evaluation of the data, reviews of the report produced from the raw data, and verification that the QC checks are within required limits (e.g. spikes, surrogates, blanks, duplicate spikes, etc.). The raw data and final report are submitted for verification.

Section D.1.2: Data Verification/Methods

The laboratory manager or designated experienced chemist verifies data is correct as reported. A manager reviews 100% of the raw data against the report (to verify data interpretation made by the chemist and that QC checks are correct) and makes sure no transposition errors were made. The laboratory QA Officer reviews a percentage of all reports to verify that data meets all requirements of the QAPP. The specific procedures to be followed by the laboratory are described in the laboratory QAM. The flags used on the data will be consistent with those used by EPA for CLP data (J, R, U, B, etc.). The laboratory stores all raw data in their archives for five years. Raw data is available to MPCA staff as needed.

The MPCA Program staff does a data review when the analytical report is received. MPCA staff review data to verify all QC is acceptable, the project requirements are met (holding times and reporting limits), and that all required information is present in the report. The MPCA project manager reviews the data to ensure that all quality control requirements are met. The project manager also reviews the field duplicates, calculates the RPD, and compares the data to past data from the site to verify consistency. When all the data points have been reviewed, the project manager compares the data which is acceptable to the data which was planned for the site and verifies that the completion rate goal has been met. Any problems with the data or laboratory issues are immediately brought to the attention of the MPCA QAC who contacts the laboratory to assess the problems and find a solution. If the problem is particularly severe, a data audit or full laboratory audit may be conducted.

Section D.1.3: Data Validation/Methods

At least 10% of the data are validated by the MPCA QA Coordinator from the raw data. The validation process is consistent with the *National Functional Guidelines for Organic Data Review*. If any data problems are identified, more data packages are validated. If data does not meet the QAPP requirements and are judged to be unusable, the analyses are not paid for and the samples are re-collected.

Section D.2: Reconciliation with User Requirements

Data quality objectives have been met when a complete report (with all data qualifiers) has been provided to the U.S. EPA. The report includes any data issues identified by the laboratory or the MPCA. The report points out any limitations on the use of the data to decision makers.

Section D.3: References

1. Minnesota Statute Chapter 115C, "Petroleum Tank Release Cleanup Act."

2. Subtitle I of the Solid Waste Disposal Act created the UST Program

3. The Energy Policy Act of 2005 (in Title XV, Subtitle B) contains new amendments for the UST Program

4. U.S. Environmental Protection Agency, 2001 (Reissued May 2006). *EPA Requirements for Quality Assurance Project Plans (QA/R-5)*, EPA/240/B-01/003, Office of Environmental Information.

5. U.S. Environmental Protection Agency, 2002. *Guidance for Quality Assurance Project Plans (QA/G-5)*, EPA/240/R-02/009, Office of Environmental Information.

6. Environmental Performance Partnership Agreement, October 1, 2016 – September 30, 2020, FFY 2017 – 2020, Minnesota Pollution Control Agency, Region 5, U. S. Environmental Protection Agency.

7. US EPA Contract Laboratory Program, June, 2008, *National Functional Guidelines for Superfund Organic Methods Data Review,* EPA-540/R-08-01, OSWER 9240.1-48, Office of Superfund Remediation and Technology Innovation.

8. US EPA Contract Laboratory Program, January, 2010, *National Functional Guidelines for Inorganic Superfund Data Review,* EPA 540-R-10-011, OSWER 9240.1-51, Office of Superfund Remediation and Technology Innovation.

Appendix 1

Table of Acronyms

CA	Corrective Action
COC	Chain of Custody
CFR	Code of Federal Register
%D	Percent Difference
DQO	Data Quality Objectives
EPA	Environmental Protection Agency
EnPPA	Environmental Performance Partnership Agreement
FOC	Field Operations Center
LIMS	Laboratory Information Management System
LUST	Leaking Underground Storage Tank
MPCA	Minnesota Pollution Control Agency
MS/MSD	Matrix Spike/Matrix Spike Duplicate
PE	Performance Evaluation (sample)
PM	Project Manager
QAC	Quality Assurance Coordinator
QAO	Quality Assurance Officer
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RSD	Relative Standard Deviation
RPD	Relative Percent Difference
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SRF	Sample Receipt Form
UST	Underground Storage Tank