

**MANUAL FOR THE CERTIFICATION OF LABORATORIES
ANALYZING ENVIRONMENTAL SAMPLES FOR
THE UNDERGROUND STORAGE TANK PROGRAM OF
THE IOWA DEPARTMENT OF NATURAL RESOURCES**

Criteria and Procedures Quality Assurance

Chapter 2 – Underground Storage Tanks

Prepared for

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INTRODUCTION

The Code of Iowa sections 455B.113 and 455B.114 require certification for laboratories performing analyses of samples which are required to be submitted to the Iowa Department of Natural Resources (DNR) as a result of Iowa Code provisions, rules, operation permits, or Administrative Orders. The following procedures and criteria for certifying laboratories analyzing environmental samples for the Underground Storage Tank Program of the DNR were developed under an agreement between the DNR and the State Hygienic Laboratory (SHL) in order to implement that certification for the underground storage tank (UST) program.

The certification program applies to laboratory analytical methods for petroleum contamination of soil and water as required in Iowa Administrative Code 567—135(455B). Other test procedures for use in the field, such as the release detection methods required in Iowa Administrative Code 567--135(455B), are not included in this certification.

The risk-based corrective action (RBCA) approach to remediation of petroleum contaminated sites in Iowa was implemented in 1997. Decisions regarding a contaminated site are based on health risks associated with the individual components of simple aromatics (benzene, toluene, ethylbenzene and total xylenes, or BTEX, which are major components of gasoline) and polycyclic aromatics (naphthalene, chrysene, benzo(a)anthracene, and benzo(a)pyrene, or PAHs, which are components of heavier products), ignoring any toxic properties of the bulk materials themselves.

APPLICATION PROCEDURE

Initial inquiries regarding certification should be made either to DNR, UST Program (mailing address on cover, or telephone 515-725-0343), or to SHL, Laboratory Extension Division (mailing address on cover, or telephone 319-335-4500). A packet of materials will be sent to the applicant including the DNR fee schedule, this manual, and the pre-survey form. The material should be reviewed and the fee form sent, with a check or money order for the required fee, along with other requested information to DNR.

Following receipt and review of the application and resolution of any apparent questions, a laboratory on-site visit by SHL personnel will be scheduled. The on-site inspection will use the criteria in this document as well as a review of the laboratory's capability of running the methodology required by Iowa Administrative Code 567--135(455B) and general quality assurance of the laboratory. For non-resident laboratories, the cost of the on-site visit is charged in addition to the fee required by DNR.

Following review of the laboratory, SHL may request certain items to be addressed prior to recommending certification. The laboratory will be given an opportunity to implement or otherwise respond to the request. SHL will then prepare a summary report of the site visit, including a recommendation for or against certification or provisional certification. The report will be submitted to DNR.

Upon receipt and review of SHL's recommendation, DNR will make a decision to issue or deny certification. A laboratory can be issued certification or provisional certification which is valid for up to a two year period. Provisional certification will be changed to full certification when all conditions of certification are met. Failure to meet all conditions of certification may result in revocation of the provisional certification.

Laboratories are required to participate, using Iowa's required methodology, in a blind proficiency testing series (PT) at least annually and must report the results of this proficiency test (whether annual or more frequent) and corrective actions, if necessary, to DNR within 30 days of receipt. Acceptance limits for Iowa laboratories may be different from those of the provider of the proficiency series since uniform methodology will be used.

DNR may grant temporary certification for a period not to exceed six (6) months upon submission of the appropriate fees and a complete and accurate pre-survey form indicating a laboratory performs the required methodology satisfactorily and can be expected to meet certification requirements. Such temporary certification shall be granted only in cases where completion of the standard certification process may be delayed more than 60 days or other extenuating circumstance to be decided by DNR.

EVALUATION CRITERIA

The following evaluation criteria are adapted from US EPA's "Manual for the Certification of Laboratories Analyzing Drinking Water--Criteria and Procedures, Quality Assurance", 5th Ed., March 2005, and Iowa Administrative Code 567--135(455B) and the methodology required therein (Table 1).

1. Personnel

1.1 Director. A laboratory's volume and scope of services may not require this position. However, there must be a person either in this position or an individual available for consultation meeting the same requirements as the Director. If the Director is also a supervisor, the requirements of 1.2 are also to be met.

1.1.1 Academic training: Minimum bachelor's degree in science is required. If bachelor's degree is in a field other than chemistry, the individual should have the number of credit hours in chemistry equivalent to a minor in chemistry.

1.1.2 Experience: Minimum of 2 years of experience in an environmental laboratory is required.

1.2 Supervisor. Minimum requirements for the supervisor position are listed below. If the supervisor is also an instrument operator, the requirements of 1.3 are also to be met.

1.2.1 Academic training: Bachelor's degree in science that includes the number of credit hours in chemistry courses required for a major in chemistry.

1.2.2 Experience: Minimum of 1 year experience in chemical analysis of environmental samples is required.

1.3 Instrument Operators. Operators for the gas chromatographs or gas chromatograph/mass spectrometers and associated data systems are required to meet the following minimum standards.

1.3.1 Academic training: Bachelor's degree in chemistry or related field. The analyst need not have a bachelor's degree if the immediate supervisor has a bachelor's degree in chemistry or related field or if the analyst has the number of credit hours in chemistry courses required for a major in chemistry.

1.3.2 Specialized training: If GC/MS instrumentation is used, satisfactory completion of a short course in GC/MS offered by equipment manufacturer, professional organization, university, or other qualified training facility is essential for these operators. Specialized training for other instruments is recommended.

1.3.3 Experience: Minimum of six months experience in the operation of GC equipment for GC operators. Minimum of 12 months experience in the operation of the GC/MS for GC/MS operators.

1.3.4 Initial qualification: After appropriate training, it is essential that the analyst demonstrate acceptable results in the analysis of an applicable set of four QC or a single PT sample.

1.4 Other Analysts. The following are required minimum standards for the other analyst position.

1.4.1 Academic training: Minimum of high school diploma or equivalent.

1.4.2 Initial qualification: After being trained in a methods training course or by a fully qualified analyst, the person being trained shall demonstrate acceptable results in the analysis of an applicable set of four QC or a single PT sample.

1.5 Analysts and Operators in Training. Data produced by analysts and instrument operators while in the process of obtaining the required training or experience are acceptable when reviewed and verified by a fully qualified analyst or the laboratory supervisor.

1.6 Waiver of Academic Training Requirement. DNR may waive the need for specified academic training, on a case-by-case basis, for highly experienced analysts.

1.7 Quality Assurance Officer. It is recommended laboratories have a designated quality assurance officer with a minimum of a bachelor's degree in science and knowledge of statistics and quality control procedures. Ideally, this position should be independent of the analytical personnel and should report directly to the laboratory director in matters relating to their function.

2. Laboratory Facilities

The laboratory facilities must be clean, have temperature and humidity adequately controlled in the instrument areas and have adequate lighting at the bench top. It is important for the laboratory to have provisions for the proper storage and disposal of chemical wastes. Exhaust hoods are required for preparation, extraction and analysis where applicable.

It is recommended a minimum of 150 to 200 square feet/laboratory person be available. It is recommended that the laboratory contain at least 15 linear feet of usable bench space per analyst. Workbench space needs to be convenient to sink, water, gas, vacuum and electrical sources free of surges. It is recommended that the organic chemical facilities be separate from other facilities. The analytical and sample storage areas must be isolated from vehicle service or parking areas, motor fuel storage, and all other potential sources of contamination.

3. Laboratory Equipment and Instrumentation

The laboratory is only required to have those instruments that are needed to perform the approved methods for which certification has been requested.

4. General Laboratory Practices

4.1 Chemicals/reagents. "Analytical reagent grade" (AR) chemicals, ACS grade or better must be used for analyses where possible. Consult Standard Methods for the Examination of Water and Wastewater", for more detailed information on reagent grades. The required UST methods, OA-1 and OA-2, require use of commercial petroleum products for standards. These must be carefully selected by the laboratory to be as representative of the product anticipated or identified as possible.

Certain common sources of standards for environmental analysis, such as Restek Corporation (110 Benner Cir, Bellefonte PA 16823, 800-356-1688), Chromatography Research Supplies (210 Fairbanks St, Addison IL 60101, 800-327-3800), Supelco (595 N Harrison Rd, Bellefonte, PA 16823 800-325-3010) or Ultra Scientific (250 Smith St, North Kingstown RI 02852, 401-294-9400), may be able to provide standards of certain commercial materials. Approved providers of blind proficiency programs (UST Soil PT Programs for BTEX, gasoline, and diesel and Water PT Programs for BTEX, gasoline, and diesel) may be found at the following link: <http://nelac-institute.org/content/NEPTP/ptproviders.php>. Mention of specific companies is for convenience and is not an endorsement of any particular company.

4.2 Laboratory safety. While specific safety criteria are not an aspect of laboratory certification unless specifically mentioned in the analytical method, laboratory personnel are expected to apply general and customary safety practices as a part of good laboratory procedure. Each laboratory is strongly encouraged to have a safety plan as part of their standard operating procedure (see OSHA requirements, especially 29 CFR 1910.1450). Where safety practices are included in a method, they must be strictly followed.

4.3 Reagent water. Reagent water for petroleum product analysis is to be free of interferences for the analytes being measured. It may be necessary to treat water with activated carbon to eliminate all interferences.

4.4 Glassware preparation. Glassware and sample bottles must be washed in a detergent solution and thoroughly rinsed, first in tap water and then in reagent water. Glassware should have a final organic solvent rinse or for non-volumetric glassware, must be baked at 400oC for 30 minutes and then dried or cooled in an area free of organic contamination. Glassware should be covered with organic-free aluminum foil during storage. Bottles and cap liners, used for collection of samples for determination of volatile organic chemicals (BTEX), must be dried at 105oC for 1 hour, sealed, and stored in an area free of volatile organics.

5. Analytical Methodology

5.1 Required methodology. Iowa Administrative Code 567--135(455B) requires use of the following methodology: Method OA-1, "Method for Determination of Volatile Petroleum Hydrocarbons (Gasoline)", revision 7/27/93, EPA methods 8015B or 8260B, SW-846, "Test Methods for Evaluating Solid Waste," current Edition; Method OA-2 "Method for Determination of Extractable Petroleum Products (And Related Low Volatility Organic Compounds)", revision 7/27/93; EPA methods 525.2, 550, 550.1 for polynuclear aromatic hydrocarbons (PAHs) in drinking water; EPA methods 525.2, 550, 550.1, 610, 8100, 8270, 8310 for PAHs in water; EPA methods 8100, 8270, 8310 for PAHs in soil; and NIOSH 1501 for BTEX in soil gas (summarized in Table I). Latest revisions of methods OA-1 and OA-2 are available from SHL or DNR. In general, all procedural steps in these methods are considered requirements. The methods are designed to determine common petroleum products stored in or otherwise involved with underground storage tanks. This includes detection of saturated hydrocarbon components that may be present as a result of spillage or leakage when other components such as aromatics,

olefins, or volatiles may have been degraded or completely removed in the environment. Other methods cannot be used without prior approval of DNR.

Method OA-1, EPA 8015B or 8260B are used principally to analyze BTEX, and OA-2 is used to provide surrogate analysis of the PAHs based on default concentrations of the PAHs in certain petroleum-derived materials, as explained in the regulations. Under RBCA rules, analysis of product gasoline (as opposed to the individual BTEX components) by OA-1 is not generally required and laboratories may not wish to continue to run proficiency samples for product; however, if product data are generated by that method to be submitted to DNR for special circumstances, the laboratory must be certified for this analysis in order for the result to be acceptable to DNR. Laboratories must run and be able to qualitatively identify gasoline by OA-2.

Laboratories must meet the Minimum Detectable Level requirements as stated in the Iowa Administrative Code for all exposure pathways as specified in the "Iowa Tier 1 Look-Up Table" in by Chapter 135.9 (1) f and Section 135.10 (3) g (5). The final cited section indicates that samples from sources within one mile of a public water supply must be at a contamination level less than 40 % of the values listed in the Tier 1 Look-Up Table.

Laboratories must participate in a blind proficiency testing program such as the AIHA/NIOSH Proficiency Analytical Testing Program using this NIOSH method 1501 for soil gas and acceptable results for the BTEX compounds on the most recent round containing those analytes. Holding time for soil gas samples in Iowa is 14 days with refrigeration and storage separate from any source of contamination.

Integration of petroleum products

The petroleum products that are the primary concern under the State of Iowa's Underground Storage Tank Program consist of a large number of different hydrocarbon components which may not be fully resolved chromatographically. Undegraded diesel fuel, fuel oil and kerosene show large, easily-resolved peaks due to selected components like the normal alkanes and a few isoprenoids, but the chromatographic profile consists of an underlying background of unresolved components which are part of the material. Generally, degraded products have reduced isolated peaks relative to the unresolved portion. The chromatographic profile of motor oil, another common pollutant, consists primarily of a broad "hump" of unresolved components. It is likely that heavier compounds like naphthalene and benzo(a)pyrene, of importance from a health effects standpoint, are part of the unresolved background. This unresolved background is, in fact, part of the material and for accurate quantitation must be included in the integration. It is important that proper integration including the unresolved components be performed by all laboratories since the integrated response will be used as a surrogate for estimation of the concentrations of the target PNA compounds under RBCA. It should also be noted that Method OA-2 is not a Carbon Range method but a Product Range (i.e. – Diesel, Fuel, Oil, Waste Oil, etc.) method so that some product range may overlap in integration.

A clean chromatographic system is needed for accurate baselining, and 'baking out' of the system may be needed, especially after running samples contaminated with very heavy products. Blanks need to be run to be sure the system has returned to an acceptable and reproducible baseline if heavily contaminated samples are encountered.

With the RBCA rules, it is important for laboratories to be able to distinguish various petroleum products in method OA-2. Laboratories should analyze samples of at least gasoline, mineral spirits, kerosene, diesel fuel, fuel oil, and motor oil at least annually on their systems and keep the resulting chromatograms for reference. These reference sample analyses must be taken through the entire extraction, concentration, and analysis procedure.

5.2 Ancillary methodology. Ancillary methodology used for analysis or confirmation of benzene, toluene, ethylbenzene and xylenes--such as EPA method 502.2--may be accepted for groundwater analysis under certain limited conditions by DNR and thus may be reviewed as well if used by the laboratory, but these methods may NOT be used as primary methods for compliance with DNR's UST regulations. In certain situations, other ancillary methods such as those used for detection of oxygenates or other additives or for GC/MS identification

of specific chemicals that may be stored in tanks may be useful for DNR's UST program; capability of the laboratory to provide these additional analytical methods may be noted also but are not certified under this certification program.

6. Sample Collection, Handling and Preservation

- 6.1** The manner in which samples are collected and handled is critical for obtaining valid data. A written sampling protocol with specific sampling instructions must be available to sample collectors and for inspection by DNR. Site assessment must be consistent with Iowa Administrative Code 567 chapter 135(455B). Chain of custody should be maintained and documented for cases that may involve legal action and is recommended as a Good Laboratory Practice for all samples taken. Laboratories not responsible for sampling should still be aware of regulatory requirements in order to properly advise their clients and evaluate possible errors in collection.
- 6.2 Rejection of samples.** The laboratory must reject any sample taken for compliance purposes not meeting the criteria in 6.2 through 6.6 below and notify the individual requesting the analyses. When analysis is performed that does not meet these criteria, the laboratory must qualify (flag) the report of results explicitly and prominently, fully disclosing the nature of the deficiency(ies) as required under Iowa Administrative Code 567-135(455B).
- 6.3 Sample containers and preservation.** The type of sample container and the required preservative are listed in the methods and summarized in Table II.
- 6.4 Maximum holding times.** Samples must be analyzed within the maximum holding times listed in the methods and summarized in Table II.
- 6.5 Sample collection and transport.** When the laboratory has responsibility for sample collection, handling, and preservation, there needs to be strict adherence to correct sampling procedures, complete identification of the sample, and prompt transfer of the sample to the laboratory. If the laboratory is not responsible for sample collection, handling, and preservation, the sample custodian must check the samples for these items to the extent possible and document violations. The report of results must disclose the nature of the deficiency (ies).
- 6.6 Sample collector.** The collector should be trained in sampling procedures and must adhere to sample collection instructions per DNR regulations.
- 6.7 Sample report form.** The sample report form must contain the information listed in the Iowa Administrative Code 567—135(455B). Indelible ink should be used.

7. Quality Assurance.

- 7.1 General requirements.** All quality control information must be available for inspection by DNR at any time or by SHL during site visits. A manual of analytical methods and the laboratory's QA plan are also to be available to the analysts.
- 7.1.1 QA Plan.** All laboratories analyzing UST compliance samples must adhere to the QC procedures specified in the methods. This is to insure routinely generated analytical data are scientifically valid and defensible and are of known and acceptable precision and accuracy. To accomplish these goals, each laboratory must prepare a written description of its quality assurance activities (a QA plan). All laboratory personnel must be familiar with the contents of the QA plan. This plan should be submitted to the auditors for review prior to the on-site visit or should be reviewed as part of the on-site visit.

The laboratory QA plan should be a separately prepared text. However, documentation for many of the listed QA plan items may be made by reference to appropriate sections of this manual, the laboratory's standard operating procedures, (SOPs) or other literature (e.g., promulgated methods). The QA Plan should be updated as necessary. At a minimum, the following items should be addressed in each QA plan:

1. Laboratory organization and responsibility
 - Include a chart or table showing the laboratory organization and lines of responsibility, including QA managers;
 - List the key individuals who are responsible for ensuring the production of valid measurements and the routine assessment of measurement systems for
 - Precision and accuracy (e.g., who is responsible for internal audits and reviews of the implementation of the plan and its requirements);

- Reference the job descriptions of the personnel and describe training to keep personnel updated on regulations and methodology, and document that
 - Laboratory personnel have demonstrated proficiency for the methods they perform.
2. Process used to identify clients' Data Quality Objectives
 3. SOPs with dates of last revision
 - Keep a list of SOPs
 - Ensure that current copies of SOPs are in the laboratory and in the QA Managers files;
 - Ensure that SOPs are reviewed annually and revised as changes are made;
 - Ensure that SOPs have signature pages and revisions dated.
 4. Field sampling procedures
 - describe the process used to identify sample collectors, sampling procedures and locations, required preservation, proper containers, correct sample container cleaning procedures, sample holding times from collection to analysis, and sample shipping and storage conditions;
 - Ensure that appropriate forms are legibly filled out in indelible ink or hard copies of electronic data are available;
 - Describe how samples are checked when they arrive for proper containers and temperature and how samples are checked for proper preservation before analysis;
 - Ensure that sampling protocol is written and available to samplers.
 5. Laboratory sample handling procedures
 - use bound laboratory note books, filled out in ink; entries dated and signed (A secure, password protected, electronic data base is acceptable);
 - store unprocessed and processed samples at the proper temperature, isolated from laboratory contaminants, standards and highly contaminated samples and, sometimes, each other; holding times may not be exceeded;
 - maintain integrity of all samples, (e.g., by tracking samples from receipt by laboratory through analysis to disposal);
 - require Chain-of-Custody procedures for samples likely to be the basis for an enforcement action;
 - specify criteria for rejection of samples which do not meet shipping, holding time and/or preservation requirements and procedures for notification of sample originators.
 6. Calibration procedures (may reference SOP)
 - specify type of calibration used for each method and frequency of use;
 - describe standards' source, age, storage, labeling;
 - perform data comparability checks;
 - use control charts.
 7. Analytical procedures (may reference SOP)
 - cite complete method manual;
 - describe quality control procedures required by the methods that must be followed.
 8. Data reduction, validation, reporting and verification (may reference SOP)
 - describe data reduction process: method of conversion of raw data to final concentrations.;
 - describe data validation process;
 - describe reporting procedures, include procedures and format;
 - describe data verification process;
 - describe procedure for data corrections.
 9. Type of quality control (QC) checks and the frequency of their use (may reference SOP)
 - instrument performance check standards;

- frequency and acceptability of method detection limit (MDL) calculations;
 - calibration, internal and surrogate standards;
 - laboratory reagent blank, field reagent blank and trip blank;
 - field and laboratory matrix replicates;
 - quality control and performance evaluation samples;
 - laboratory fortified blank and laboratory fortified sample matrix replicates;
 - initial demonstration of method capability and use of control charts;
 - qualitative identification/confirmation of contaminants.
10. List schedules of internal and external system and data quality audits and inter laboratory comparisons (may reference SOP)
 11. Preventive maintenance procedures and schedules
 - describe location of instrument manuals and schedules and documentation of routine equipment maintenance;
 - describe availability of instrument spare parts in the laboratory;
 - list any maintenance contracts in place.
 12. Corrective action contingencies
 - describe response to obtaining unacceptable results from analysis of PT samples and from internal QC checks;
 - name persons responsible for the various corrective actions;
 - describe how corrective actions taken are documented;
 13. Record keeping procedures
 - describe procedures and documentation of those procedures;
 - list length of storage, media type (electronic or hard copy);
 - describe security policy of electronic databases.

If a particular listed item is not relevant, the QA plan should state this and provide a brief explanation (e.g., some laboratories do not collect samples and thus are not required to describe sampling procedures but should be aware of DNR requirements for sampling). A laboratory QA plan should be concise but responsive to the above-listed items while remaining brief and easy to follow. Minimizing paperwork while improving dependability and quality of data are the intended goals. The QA Plan should describe how and what the laboratory is actually doing, not theory or suggested practices.

- 7.1.2 ASTM Type I, Class 1 or 2 weights or better should be available to make periodic checks on balances. A record of these checks is to be available for inspection. The specific checks and their frequency are to be as prescribed in the laboratory's QA plan and the laboratory's operations manual, if appropriate. This frequency should be at least once every 12 months.

7.2 Analytical Quality Control. The following are required for each method:

- 7.2.1 The laboratory must analyze PT samples at least annually by each method/matrix. The proficiency testing program in which the laboratory is enrolled must meet the criteria listed under "Criteria for UST Laboratory Proficiency Program" in this manual.
- 7.2.2 At least once each quarter, the laboratory must analyze a QC sample independent of the materials and preparation of calibration standards. If errors exceed limits specified, corrective action is to be taken and documented, and a follow-up quality control standard analyzed as soon as possible to demonstrate the problem has been corrected.
- 7.2.3 A standard curve composed of at least a reagent blank and three standards (six standards for NIOSH method 1501) covering the sample concentration range are to be prepared. These standards should be from a different source than the quality control standard used for 7.2.2.
- 7.2.4 At the beginning of each day that samples are to be analyzed, the standard curve is to be verified by analysis of at least a reagent blank and one standard in the expected concentration range of the samples

analyzed that day. All checks should be within +/- 20% of the standard curve or the system must be recalibrated. NIOSH method 1501 requires a calibration curve be run each day samples are analyzed.

- 7.2.5 If the reagent blank specified in 7.2.4 is not carried through the full analytical procedure, then some other blank (at least one per day) is to be carried through the entire analytical procedure. Results from reagent blanks should not exceed the laboratory's method reporting limit (see paragraph 7.2.8).
- 7.2.6 The laboratory must analyze known spikes on a regular basis at a frequency of a minimum of one sample per batch of samples of 20 samples or a minimum of one per day if batch is <20. This spike is to be analyzed through the complete analytical system. If any spike result is out of the statistical acceptance range (generally not to exceed +/- 40% for products and +/- 20% for BETX) except for a demonstrated matrix effect in the case of a matrix spike, corrective action is to be taken in accordance with the laboratory's QA plan.
- 7.2.7 The laboratory must calculate traditional control limits on an on-going basis for each analyte. The laboratory may use quality control criteria in the sections above more stringent than those stated, if their experience with on-going analytical operations demonstrates such limits to be appropriate for their operations.
- 7.2.8 It is further recommended the laboratory calculate the MDL at least annually in accordance with the procedure given in 40 CFR Part 136, Appendix B.
- 7.2.9 A laboratory must demonstrate adequate chromatography to distinguish common petroleum products such as gasoline by OA-1 and at least gasoline, mineral spirits, kerosene, diesel fuel/fuel oil, and motor oil by OA-2, maintaining a file of example chromatograms of known materials as a part of the methods as implemented at the laboratory.

8. Records and Data Reporting

8.1 Laboratory Records. Records of chemical analyses are to be kept by the laboratory for a minimum of 5 years. This includes all raw data, calculations, and quality control data. These data files may be either manual or computer based.

8.2 Data Reporting. As required by Iowa Administrative Code 567--135(455B) all laboratory reports must contain the following information:

- a. Laboratory name, address, phone number and DNR Lab Number.
- b. Medium sampled (soil, water).
- c. Client submitting sample (name, address, phone number).
- d. Sample collector (name, phone number).
- e. UST site address.
- f. Client's sample location identifier.
- g. Date sample was collected.
- h. Date sample was received at laboratory.
- i. Date sample was analyzed.
- j. Results of analyses and units of measure.
- k. Detection limits.
- l. Methods used in sample analyses (preparation method, sample detection method, and quantitative method).
- m. Laboratory sample number.
- n. Analyst name.
- o. Signature of analyst's supervisor.
- p. Condition in which the sample was received at the laboratory and whether it was properly sealed and preserved. For example, pH should be noted on the report.
- q. Note that analytical results are questionable if a sample exceeded an established holding time or was improperly preserved. The holding time for properly cooled and sealed petroleum contaminated samples is 14 days, except for water samples containing volatile organic compounds which have a 7-day holding time unless acid preserved.
- r. Laboratory reports required by this chapter for tank closure investigations under 135(455B) and site checks or Tier 1 or Tier 2 assessments must include a copy of the analytical results and chromatograms for the waste oil, diesel and gasoline standard used by the laboratory in analyzing submitted samples, properly

identified as to the material and with BTEX peaks labeled (if this information is not on the chromatograms, the portion of the associated quantitation reports with the information should be included). In addition to the analytical result, the laboratory analytical report for each sample must state whether the sample tested matches the laboratory standard for waste oil, diesel, or gasoline or that the sample cannot be reliably matched with any of these standards. A copy of the identified and labeled chromatograms (or chromatograms and associated quantitation reports if the latter are needed to provide the identification and peak labeling) for only the soil and groundwater samples with the maximum concentrations of BTEX and TEH must be included.

- s. If analyses are performed by a subcontracted laboratory, the report to DNR must include the Iowa laboratory certification number for and analytical result form from that laboratory.

REQUIREMENTS FOR MAINTAINING CERTIFICATION

To maintain UST laboratory certification in Iowa, laboratories must meet the following requirements:

- a. Laboratories must use the approved methodology for all UST analyses to be submitted to DNR;
- b. Certified laboratories must satisfactorily analyze PT samples at least once annually by each method and in all matrices. Results must be submitted to DNR or SHL along with a statement of method used within 30 days of receipt from the supplier (Note: most PT vendors will provide this data directly to DNR and this is the preferred method of delivery. Please check DNR on your submittal record with your PT provider);
- c. Laboratories must notify DNR or SHL in writing within 15 days of major changes in personnel, equipment, laboratory facilities, or other change which might impair analytical capability.
- d. Laboratories must agree to a periodic site visit, normally at least every two years. However, an on-site inspection may be conducted more frequently if the laboratory undergoes a major change or fails a PT sample, or if DNR questions an aspect of data submitted which is not satisfactorily resolved.

CRITERIA AND PROCEDURE FOR DOWNGRADING/REVOKING CERTIFICATION STATUS

Criteria for Downgrading Certification Status. A laboratory will be downgraded to provisionally certified status for any of the following reasons:

- a. Failure to analyze a PT sample annually within the Iowa acceptance limits;
- b. Failure of a certified laboratory to notify DNR within 15 days of major changes which might impair analytical capability; or
- c. Failure to satisfy DNR that the laboratory is maintaining the required standard of quality based on an on-site evaluation.

Procedure for Downgrading to Provisionally Certified Status. If a laboratory is subject to downgrading on the basis of the indicated criteria, DNR will notify the laboratory director or owner, in writing. The laboratory director will review the problems cited and, within 30 days of receipt of the letter, send a letter to DNR specifying what corrective actions are being taken. DNR will consider the adequacy of the response and notify the laboratory by mail of its certification status and may follow up to insure corrective actions have been taken.

If a laboratory fails to analyze an unknown test sample within the acceptance limits, DNR will not downgrade certification if the laboratory identifies and corrects the problem to DNR's satisfaction within 30 days of being notified of the failure. SHL may send the laboratory another unknown sample containing the failed component to verify correction or to confirm adequacy of data if no cause was unequivocally found. If the laboratory fails to analyze this second unknown sample within acceptance limits, DNR will downgrade the laboratory to provisional certification and notify the laboratory in writing.

During any phase of this procedure, a laboratory may request SHL to provide technical assistance to help identify and resolve any problem.

Once DNR notifies a laboratory, in writing, that it has been downgraded to "provisional certification" the laboratory must correct its problem within 3 months for a procedural, administrative or minor equipment deficiency and 6 months for a major equipment deficiency. If the laboratory was downgraded because of a failure to analyze a PT sample within

the acceptance limits, the laboratory must correct its problems and satisfactorily analyze another PT sample within 2 months of being notified.

Criteria for Revoking Certification Status. Laboratory certification will be revoked for the following reasons:

- a. For provisionally certified laboratories, failure to analyze a PT sample within the Iowa acceptance limits;
- b. Failure to satisfy DNR that the laboratory has corrected deviations identified during the on-site evaluation within 3 months for a procedural, administrative or minor equipment deficiency or 6 months for a major equipment deficiency;
- c. Submission of a PT sample to another laboratory for analysis;
- d. Falsification of data or other deceptive practices; or
- e. Failure to use required analytical methodology for UST analyses submitted to DNR.
- f. Failure to satisfy DNR that the laboratory is maintaining the required standard of quality based on an on-site evaluation.
- g. Loss of certification in resident state if certified via reciprocally.

Procedure for Revoking Certification. DNR will notify the laboratory of the intent to revoke certification by commencement of a contested case proceeding as provided in 561 IAC 7.5(2) and consistent with Code 17A.18. Certification will be reinstated when and if the laboratory can demonstrate that all conditions of laboratory certification have been met through a new application for certification.

CRITERIA FOR UST LABORATORY PROFICIENCY PROGRAM

Laboratories must be enrolled in a proficiency testing program to receive certification under Iowa Department of Natural Resources (DNR) regulations for the Underground Storage Tank (UST) regulations. To DNR’s knowledge, a proficiency program administered by the USEPA or other government agency does not exist. Thus, third-party testing programs must be used. To assure the adequacy of the evaluation of laboratory performance, the following criteria must be met by the third-party proficiency testing program.

1. The organization conducting the testing must be an independent proficiency testing organization not subject to influence by the laboratories enrolled in the proficiency testing program. The proficiency testing organization must have set QA procedures to ensure the validity of the test samples. A list of approved PT programs may be found at the following link - <http://nelac-institute.org/content/NEPTP/ptproviders.php>.
2. The program must be blind to the participating laboratories; that is, the true values, expected ranges other than very general orders of magnitude, or other identification that would provide assistance to a laboratory in determining the result must be unknown to the laboratory and remain so until past a cut-off date beyond which no results will be accepted by the proficiency testing organization.
3. The range of proficiency samples provided must include both gasoline and BTEX (benzene, toluene, ethylbenzene and xylene(s)) for analysis by OA-1 and one or more of the following for analysis by OA-2: mineral spirits, kerosene, diesel fuel, fuel oil, or motor oil. Both soil and water matrices must be used for these products as summarized in the table below.

METHOD	MATRIX	PRODUCT SPIKED	CONC. RANGE
OA-1	Soil	Gasoline	1-2000 mg/kg
OA-1	Water	Gasoline	0.5-100 mg/L
OA-1	Soil	BTEX	0.25-10 mg/kg
OA-1	Water	BTEX	0.005-1 mg/L
OA-2	Soil	Diesel fuel or related	1000-4000 mg/kg
OA-2	Water	Diesel fuel or related	0.5-100 mg/L
various	D Water	PAHs	as determined by third party
Various	Water	PAHs	as determined by third party
various	Soil	PAHs	as determined by third party
1501	Air	BTEX	as determined by third party

The concentration ranges listed are advisory to indicate the range where proficiency should be demonstrated; proficiency testing organizations are not necessarily bound to provide all PT samples within that range but should have at least some within it. At least one proficiency testing round per year must be conducted; many laboratories may wish more frequent rounds or the availability of remedial rounds.

- Results of each proficiency testing round must be available by the proficiency testing organization within 90 days of issuance of the round. The report of results from the proficiency testing organization must give the “true value” along with the laboratory’s reported analysis. The state of Iowa requires laboratory results to be within the following acceptance ranges (percentages) of the true value for proficiency to be demonstrated, regardless of the acceptance range established by the proficiency testing organization.

METHOD	MATRIX	PRODUCT SPIKED	ACCEPTANCE RANGE
OA-1	Soil	Gasoline	+/- 40%
OA-1	Water	Gasoline	+/- 40%
OA-1	Soil	BTEX	+/- 20%
OA-1	Water	BTEX	+/- 20%
OA-2	Soil	Diesel fuel or related	+/- 40%
OA-2	Water	Diesel fuel or related	+/- 40%
various	D Water	PAHs	as determined by third party
Various	Water	PAHs	as determined by third party
various	Soil	PAHs	as determined by third party
1501	Air	BTEX	as determined by third party

The proficiency testing organization should provide the results for any laboratory that requests it directly to the DNR, as well as to the submitting laboratory. However, it is the responsibility of the enrolled laboratory(ies) to ensure that the proficiency testing organization submits the results to DNR or otherwise transmits the results to DNR within 30 days of receipt.

Table 1 Approved Methods

Contaminant	Matrix	EPA	Other
BTEX	Water	8015C, 8260B	OA-1
BTEX	Soil	8015C, 8260B	OA-1
Volatile Petroleum Hydrocarbons	Water	8015C, 8260B	OA-1
Volatile Petroleum Hydrocarbons	Soil	8015C, 8260B	OA-1
Total Extractable Hydrocarbons	Water	8015C*	OA-2
Total Extractable Hydrocarbons	Soil	8015C*	OA-2
PAHs	Drinking water	525.2, 550, 550.1	
PAHs	Water	525.2, 550, 550.1, 610, 8100, 8270, 8310	
PAHs	Soil	8100, 8270, 8310	
BTEX	Soil gas		NIOSH 1501

BTEX: Benzene, Toluene, Ethylbenzene, Xylene (total)

PAHs: Naphthalene, Benzo(a)pyrene, Benz(a)anthracene, Chrysene

*Note – Carbon Range Method for quantitation specified in SW-846 Method 8015 is not applicable. Concentrations should still be calculated using the retention time range of individual petroleum products.

Table 2 Preservation and Hold Times

Parameter / Method	Preservative (Soil and Water)	Sample Holding Time	Suggested Sample Size	Type of Container
OA-1	HCl for aqueous, hermetic seal with no headspace, Cool 4°C	14 days collect → analysis 7 days if not acidified	40 ml or 100 g, collect in duplicate	Glass with teflon lined septum
OA-2	Cool 4°C	40 days extract → analysis water - 7 days collect → extract soil - 14 days collect → extract	1 L or 100 g	Glass with teflon lined septum
525.2	Sodium sulfite, HCl pH<2, Cool 4°C	14 days collect → extract 30 days collect → analysis	1L or 1quart	Amber glass with teflon lined septum
550	Sodium thiosulfate and HCl pH <2 (if chlorine is present), Cool 4°C	7 days collect → extract 30 days extract → analysis	1L or 1 quart	Amber glass with teflon lined septum, or protect from light if not Amber
550.1	Sodium thiosulfate and HCl pH <2 (if chlorine is present), Cool 4°C	7 days collect → analysis 40 days collect → analysis	1L or 1 quart	Amber glass with teflon lined septum
610	Sodium thiosulfate, Cool 4°C, store in dark	7 days collect - extract 40 days extract → analysis	1 L or 1 quart	Glass with teflon lined cap
8100	Cool 4°C (if chlorine present, sodium thiosulfate)	40 days extract → analysis water - 7 days collect → extract soil - 14 days collect → extract	1 L or 100 g	Glass with teflon lined septum, amber
8015	Cool 4°C, sodium thiosulfate if chlorine present	40 days extract → analysis water - 14 days collect → extract soil - 14 days collect → extract		Glass with teflon lined septum, amber
8260	Cool 4°C, sodium thiosulfate if chlorine present	40 days extract → analysis water - 14 days collect → extract soil - 14 days collect → extract		Glass with teflon lined septum, amber
8270	Cool 4°C, sodium thiosulfate if chlorine present	40 days extract → analysis water - 14 days collect → extract soil - 14 days collect → extract	1 L or 100 g	Glass with teflon lined septum, amber
8310	Cool 4°C, sodium thiosulfate if chlorine present	40 days extract → analysis water - 7 days collect → extract soil - 14 days collect → extract	1 L or 100 g	Glass with teflon lined septum, amber
1501	Cool 4°C, recommended	14 days collect → analysis	Maximum volume = 200 ml	glass tube, 7 cm long, 6 mm OD, 4 mm ID Glass with teflon lined septum

**CERTIFICATION OF LABORATORIES ANALYZING
UST SAMPLES
FOR THE IOWA DEPARTMENT OF NATURAL RESOURCES**

PRESURVEY INFORMATION

The Iowa DNR will be launching a web application in early 2019 where many of these questions can be answered online and documents can be attached electronically. Please check our website at www.iowadnr.gov/labcert.

Please fill in appropriate information relevant to solid waste analyses only. Attach copies of the following documents, as available and applicable to these analyses:

- Quality Assurance Plan
- Sampling Instructions and Forms
- Example Analysis Report
- Proficiency Testing Program Results

Fill in appropriate information for those items for which certification is requested.

Return forms, fees, and all material requested to:

Iowa Department of Natural Resources
Laboratory Certification
6200 Park Ave Ste 200
Des Moines IA 50321

ANALYTICAL METHODOLOGY

Attach a list of analytical methods used and their dates/revision numbers.

SAMPLE COLLECTION, HANDLING AND PRESERVATION

Attach copies of sampling instructions and forms provided to samplers.

Briefly describe sample storage facilities:

QUALITY ASSURANCE

Attach a copy of the laboratory's QA plan.

Analytical quality control

Blind Performance evaluation programs relevant to solid waste analyses that the lab participates in:

Submit copies of results in the last year along with identification of the methodology used for analysis of the PE sample.

Source of and frequency of external check samples for each method:

Frequency and type of blanks, duplicates and spikes for each method:

RECORDS AND DATA REPORTING

How long are records, including raw data, kept? _____

Include a copy of an example analysis report of a solid waste sample.