

**R211 – SPECIFIC REQUIREMENTS:**  
**WYOMING STORAGE TANK REMEDIATION TESTING LABORATORY**  
**ACCREDITATION PROGRAM**

(Formerly known as Wyoming Remediation of Leaking Aboveground and  
Underground Storage Tank (LAUST) Laboratory Accreditation Program)

February 2008

Cover Page

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LABORATORY ACCREDITATION PROGRAM

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R211 – SPECIFIC REQUIREMENTS: WYOMING STORAGE TANK REMEDIATION  
TESTING LABORATORY ACCREDITATION PROGRAM

**1. Introduction**

This document describes the accreditation requirements applicable to the State of Wyoming, Department of Environmental Quality, Solid & Hazardous Waste Division (WDEQ/SHWD) Storage Tank Remediation (STR) Program Guidance Document #8. Laboratories that are not accredited through this program may not perform work for the WDEQ/SHWD STP.

This document details the requirements for accreditation and the parameters and methods on which accreditation will be granted. This requirements document will assure that laboratories performing analytical testing relating to the STR have the technical proficiency, competency, staffing, and management to properly perform analytical testing.

The International Organization for Standardization/International Electro-technical Commission (ISO/IEC) ISO/IEC 17025: 2005, the American Association for Laboratory Accreditation (A2LA) Environmental Program Requirements, and State requirements form the basis for accrediting laboratories for the STR.

**Scope** A2LA will accredit environmental laboratories on an analyte-specific basis. Laboratories must be able to demonstrate that they are accredited by A2LA for compliance with the following parameters and one or more of the following test methods for each parameter:

<u>Parameter</u>	<u>EPA Method Identification</u>
<b>Metals</b>	
Cadmium	6010C, 7131A
Chromium (total, hexavalent, and trivalent)	6010C, 7196A
Lead	6010C, 7421
<b><u>Purgeable Organics (volatiles)</u></b>	
Benzene	5030B/8021B, 5030B/8260B
Diisopropyl Ether	5030B/8021B, 5030B/8260B
Ethyl benzene	5030B/8021B, 5030B/8260B
Ethyl <i>tert</i> -Butyl Ether	5030B/8021B, 5030B/8260B
Gasoline Range Organics C <sub>6</sub> -C <sub>10</sub>	5030B/8260B, 8015C
Methyl <i>tert</i> -Butyl Ether	5030B/8021B, 5030B/8260B
Naphthalene	5030B/8021B, 5030B/8260B
<i>tert</i> -Amyl Methyl Ether	5030B/8021B, 5030B/8260B
<i>tert</i> -Butyl Alcohol	5030B/8021B, 5030B/8260B
Toluene	5030B/8021B, 5030B/8260B
TPH(GRO)	8015C, 5030B/8260B (C <sub>6</sub> to C <sub>10</sub> )
Xylenes, total	5030B/8021B, 5030B/8260B
1,2-Xylene	5030B/8021B, 5030B/8260B
1,3-Xylene	5030B/8021B, 5030B/8260B
1,4-Xylene	5030B/8021B, 5030B/8260B
<b><u>Extractable Organics</u></b>	
Diesel Range Organics C <sub>10</sub> -C <sub>32</sub>	8015C with 3630, 5030B/8260B with 3630
TPH(DRO)	8015C with 3630, 5030B/8260B (C <sub>10</sub> to C <sub>32</sub> ) with 3630

The following requirements apply to the table above:

- (a) When analyzing for dissolved cadmium, Method 6010B may be used, provided the detection limit is as low as the action level for groundwater samples and at least as low as the detection limit for the 7000 series for soils.
- (b) When analyzing for benzene, ethylbenzene, naphthalene, toluene, and xylenes, Method 8021B may only be used during routine monitoring events.
- (c) Constituent analysis of benzene, ethylbenzene, naphthalene, toluene, and xylenes by Method 8260B are required for all subsurface investigation work, additional site assessments, and confirmation sampling for project closure.
- (d) When analyzing groundwater for dissolved cadmium, dissolved chromium, and dissolved lead, samples that **cannot** be delivered to the laboratory within 48 hours must be filtered onsite with a 45 micron filter. **After** the samples have been filtered, add nitric acid onsite to lower the sample pH to <2. If the samples **can** be delivered to the laboratory within 48 hours, do **not** filter the samples or acidify the samples during onsite collection.

Assessors evaluate a laboratory for compliance with the United States Environmental Protection Agency (EPA) accepted methods. In addition, a laboratory must meet the minimum requirements specified in this document.

If the laboratory is a corporation, the corporation shall be registered with the Secretary of State, State of Wyoming, and shall be authorized to do business with the State of Wyoming.

## **2. Certification Requirements**

To be certified by the STP to perform analytical testing relating to the Program, laboratories must show current accreditation by A2LA for the "Wyoming STR Program." Laboratories seeking accreditation for the STR program should contact A2LA.

Certification shall be valid for two (2) years from the date of accreditation by A2LA. Certification may be renewed with updated proof of accreditation from A2LA.

Certification may be revoked or suspended by the Program for any of the following:

- (a) The applicant negligently, incompetently, recklessly, or intentionally violates any provision of these requirements or any state, federal, or local statute, regulation, or code or standard concerning the performance of analytical testing.
- (b) The applicant obtains the certification through fraud or misrepresentations.
- (c) The applicant knowingly or intentionally submits false information to contractors or the Program.
- (d) The applicant loses its A2LA accreditation for one or more of the parameters and test methods specified in this requirement document (see Scope, above).
- (e) The applicant receives an unsatisfactory contract performance review from the WDEQ Project Manager.

The WDEQ Director shall have the authority to suspend or revoke a certification. The WDEQ Director shall then cause a letter to be issued notifying the certified laboratory of the action.

### **3. General and Specific Criteria**

The general criteria for accreditation are contained in ISO/IEC 17025:2005. All provisions of the general criteria shall apply.

Specific criteria are an elaboration on or interpretation of the general criteria plus those requirements of accreditation applicable to a certain field of testing, matrix type, testing technology, type of test, or specific test. The specific criteria applicable to the Environmental Field of Testing and Wyoming STR requirements are presented in the following sections. The numbering system for each section below corresponds to the requirement sections of ISO/IEC 17025:2005.

### **4. Management Requirements**

#### **4.1 Organization**

(No Additions)

#### **4.2 Quality System**

(No Additions)

#### **4.3 Document Control**

(No Additions)

#### **4.4 Review of Requests, Tenders and Contracts**

(No Additions)

#### **4.5 Subcontracting of Tests and Calibrations**

(No Additions)

#### **4.6 Purchasing Services and Supplies**

(No Additions)

#### **4.7 Service to the Client**

(No Additions)

#### **4.8 Complaints**

(No Additions)

#### **4.9 Control of Nonconforming Testing**

(No Additions)

#### **4.10 Improvement**

(No Additions)

#### **4.11 Corrective Action**

4E.11.1 The laboratory shall document, investigate, and take corrective action for all episodes where QC data show an out-of-control situation. The laboratory shall keep records of all out-of-control events, the determined cause(s), and corrective actions taken. All reported data from an out-of-control event shall be appropriately qualified.

#### **4.12 Preventive Action**

(No Additions)

#### **4.13 Control of Records**

The laboratory shall establish and maintain a records system ensuring that:

4E.13.1 All observations and calculations are recorded in a permanent manner (such as laboratory notebooks, pro-forma work sheets, or magnetic media) at the time they are made and that the units of measurement in which observations are recorded are stated.

4E.13.2 Original records are uniquely identified and traceable to the test items to which they refer and to any test reports based upon them.

4E.13.3 Records are traceable, retrievable, and legible and include sufficient information and explanation such that they can be readily interpreted by staff other than those responsible for their generation.

4E.13.4 Records contain sufficient information to permit identification of possible sources of error and to permit, where feasible and necessary, satisfactory repetition of the test under the original conditions.

4E.13.5 Records contain sufficient details of any significant departures from test specifications or other specified procedures including authorizations for such departures.

4E.13.6 Records are checked for data transcription or calculation errors and the checks are documented.

4E.13.7 Records identify the person or persons responsible for their creation and the date of such creation and for the person(s) checking data transcriptions and calculations and the date of such checking.

4E.13.8 Corrections or amendments to test records are made in a manner that does not obliterate the original data and are signed or initialed and dated by the person responsible.

4E.13.9 A list of all staff documenting their initials and/or signatures as used in documents such as logbooks shall be maintained.

4E.13.10 Test records shall be protected from loss, damage, misuse, or deterioration and shall be retained for an appropriate period in a manner that permits retrieval when required. Test records that are created and/or retained on magnetic media (e.g., computer disks) or photographic media (e.g., microfiche) shall be stored in a manner that protects them from the hazards that degrade such media. Provision shall be made for the printing of such records when required.

Note: Appropriate records retention periods are based upon the laboratory's various types of work and its legal and contractual obligations.

#### **4.14 Internal Audits**

(No Additions)

#### **4.15 Management Reviews**

(No Additions)

### **5. Technical Requirements**

#### **5.1 General**

(No Additions)

#### **5.2 Personnel**

5E.2.1 Technical Manager. The technical manager (however named) shall possess a 4-year college degree from an accredited educational institution in chemistry or a related science or equivalent and have at least 3 years of non-academic laboratory experience. The technical manager shall be on-site at least 50% of the time.

5E.2.2 Quality Manager. The quality manager (however named) shall possess a 4-year college degree from an accredited educational institution in a basic or applied science or equivalent and have at least 1 year of non-academic laboratory experience and training in statistics. Alternatively, the quality manager can have a college degree in other than the basic or applied sciences, with at least 4 years of non-academic analytical chemistry experience and training in statistics. The technical manager may also function as the quality manager so long as he/she does not act in the position as the sample analyst/technician analyzing the samples or act as the immediate supervisor of the analyst/technician involved with the analysis of the samples. The quality manager may be employed by the laboratory on a part-time basis or as a consultant.

5E.2.3 Senior Technical Staff. Persons in each senior technical position shall have a bachelor's degree in a relevant scientific field or equivalent experience. At least one year of non-academic experience in relevant analysis is required. Successful training in specific methods used in the laboratory shall be verified and documented as performance evaluations using reference/control materials of the matrices of concern. Proficiency testing results must be documented.

Persons filling the following job functions must meet the associated minimum experience and training requirements:

5E.2.3.1 Inductively coupled plasma-emission (ICP) spectroscopy: One year experience with satisfactory completion of a short course on ICP or an equivalent in-house training course.

5E.2.3.2 Flameless atomic absorption spectroscopy: One year with satisfactory completion of a short course on graphite furnace atomic absorption (GFAA) or an equivalent in-house training course.

5E.2.3.3 Flame atomic absorption (FLAA) spectroscopy: One year with satisfactory completion of a short course on FLAA or an equivalent in-house training course.

- 5E.2.3.4 X-ray fluorescence (XRF) spectroscopy: One year with satisfactory completion of a short course on XRF or an equivalent in-house training course.
- 5E.2.3.5 Gas chromatography: One year with satisfactory completion of a short course on basic GC or an equivalent in-house training course.
- 5E.2.3.6 Mass spectrometry: One year with satisfactory completion of a vendor's training course, professional sponsored short course, or equivalent in-house training course.
- 5E.2.3.7 Mass spectra interpretation: One year with satisfactory completion of a vendor's training course, professional sponsored short course, or equivalent in-house training course.
- 5E.2.3.8 General chemistry and instrumentation: Six months.
- 5E.2.3.9 Field testing: Six months.
- 5E.2.3.10 Sample collection: Six months.
- 5E.2.4 Analyst/Technician. Analysts/technicians shall have completed a training course (or an equivalent in-house course) in relevant analyses and have demonstrated ability to produce reliable results through accurate analysis of reference materials (RMs), proficiency testing samples, or in-house quality control samples. Their performance must be documented. Junior staff (non-degreed personnel with less than 3 years relevant experience) must work under the direct supervision of the technical manager, or under the supervision of a senior technical person described above, or under the instruction of an analyst/technician who has performed successfully over a period of three years in the relevant analyses using the same technologies being applied for the analysis of environmental samples.

5E.2.5 Minimum Level of Experience Required for Independent Operation.

- 5E.2.5.1 Sample preparation: 3 months per method used.
- 5E.2.5.2 Routine sample analysis: 6 months per method used.

5E.2.6 Requirements during Training. Analyst/technicians in training may perform work on samples submitted for environmental analysis as long as the following conditions are met:

- 5E.2.6.1 They have demonstrated the ability to produce reliable results through accurate analysis of RMs, proficiency testing samples or in-house quality control samples; and
- 5E.2.6.2 Their immediate supervisor or instructor is readily available in their work area when they are preparing and/or analyzing the samples.
- 5E.2.7 The laboratory shall have documented evidence contained in their training records of proficiency of all personnel for each test method or activity performed on the matrices of concern.

**5.3 Accommodation and Environmental Conditions**

5E.3.1 Quality Issues. In order to prevent contamination of samples or standards, the laboratory shall:

- 5E.3.1.1 Use distilled/demineralized water that is demonstrated to be free of interferants at applicable detection limits.
- 5E.3.1.2 Check and record the conductivity of distilled/demineralized water at least once a week using a

calibrated conductivity meter that is external to the water system. The point of water collection shall be from a frequently used access point.

- 5E.3.1.3 Exhaust hoods shall be vented in such a manner to prevent cross contamination of samples and equipment. (Example: The hood outlet of the organic extraction laboratory should not be near the air intake for the volatile organic laboratory. Evidence can be obtained by examination of laboratory blanks for contamination.)
- 5E.3.1.4 Provide sample storage facilities that prevent cross contamination of samples with standards, solvents, or reagents. See section 11E4 of this criteria. (Evidence of compliance may be obtained through the examination of storage areas or through the use of laboratory storage blanks, travel/trip blanks, or other blanks stored with samples.)
- 5E.3.2 Laboratory Safety: Each laboratory shall have a safety and chemical hygiene plan (see OSHA rule 29 CFR 1910) as part of its standard operating procedure. Where safety practices are included as part of an approved method, the practices shall be strictly followed. While more specific safety criteria are not an aspect of this accreditation program, laboratory personnel should apply general and customary safety practices as a part of good laboratory procedures. The laboratory shall:
  - 5E.3.2.1 Have toxic chemical handling areas consisting of impervious, non-reactive material covered with absorbent material.
  - 5E.3.2.2 Provide exhaust hoods for personnel protection (see 29 CFR 1910.1450, Occupational Exposure to Toxic Substances in Laboratories and ANSI/AIHA Z9.5-1992, Standard for Laboratory Ventilation).
  - 5E.3.2.3 Have procedures and facilities for handling material that may transmit infectious agents (see NIH 88-8395 or equivalent).
  - 5E.3.2.4 Store reagents, corrosives, explosives, oxidants, and flammable solvents appropriately (see OSHA rule 29 CFR 1910.1450).
- 5E.3.3 Waste Disposal: Each laboratory shall have waste collection, storage, and disposal procedures and policies (reference: 40 CFR 261, 262, and 264) as part of its standard operating procedures. Where disposal practices are included as part of an approved method, these practices shall be strictly followed. While more specific disposal criteria are not an aspect of this accreditation program, the laboratory should apply appropriate federal, state, and local disposal practices as a part of good laboratory procedures.

## **5.4 Test Methods and Method Validation**

The laboratory shall:

- 5E.4.1 Have documented procedures for making and controlling revisions to in-house SOPs (use revised SOPs only after written authorization by senior technical personnel).
- 5E.4.2 Have documented procedures for data collecting and reducing, reporting, and record keeping.
- 5E.4.3 Have documented method performance procedures to apply at appropriate levels of all measurement systems.

- 5E.4.4 Have documented procedures to verify test reports.
- 5E.4.5 Have documented procedures for correcting erroneously reported results.
- 5E.4.6 Prepare analytical standards at a frequency consistent with method requirements and good QC (frequency is a function of concentration and type of matrix; generally, the lower the concentration the less stable the standard).
- 5E.4.7 Specify the methods and do routine analyses for checking all solvents and reagents used for dilutions and extractions.
- 5E.4.8 SOPs for test methods shall supply or refer to information addressing the following areas:

Interferences	Instrument Calibration
Safety Considerations	Quality Control Procedures
Apparatus and Equipment	Detailed Step-by-Step Procedure
Reagents and Supplies	Sample Calculations
Sample Preservation and Storage	Method Performance Criteria
Sample Preparation	(accuracy and precision)

- 5E.4.9 Acceptable Methodology. Procedures published by federal agencies (e.g., USEPA, NIOSH), nationally or internationally recognized technical authorities, or other validated procedures may be acceptable to use once the laboratory has demonstrated adequate performance with the method for each particular matrix. Alternative procedures and/or modifications of methods may be used if they have been EPA or State approved.
- 5E.4.10 Calibration range. Linear calibration ranges (or working calibration ranges) shall be established and routinely verified for each method.
- 5E.4.11 Method detection limits. Method detection limits (MDLs) shall be established and statistically verified at least annually where appropriate for each method and matrix of concern.
  - 5E.4.11.1 For methods with stated MDLs, the laboratory shall demonstrate and document its ability to achieve such MDLs.
  - 5E.4.11.2 MDLs shall be determined using procedures published or recognized by USEPA. An example of an acceptable recommended procedure is in 40 CFR Part 136, Appendix B.

## **5.5 Equipment**

For analytical balances/pan balances:

- 5E.5.1 Analytical balances shall be capable of weighing to 0.1 mg.
- 5E.5.2 Records of balance calibration shall be kept covering at least the effective range of its use traceable to Class 2 or 3 (per ASTM E617) reference weights (formerly classified as Class S and S-1).
- 5E.5.3 Records showing functional/calibration checks for each day of use for analytical balances and monthly for pan balances shall be maintained. (Note: Criteria for the accuracy of balances must be established. ASTM E200-91 recommends an accuracy of 0.1% or less.)
- 5E.5.4 The balances shall be serviced and calibrated at least annually.  
Please refer to P102 - A2LA Policy on Measurement Traceability.

5E.5.5 The reference weights shall be calibrated at least every five years.

For pH meters:

5E.5.6 The laboratory shall use a clean pH meter with appropriate electrode with scale graduations at least 0.1 pH units (calibrated to  $\pm 0.1$  pH units for each use period).

5E.5.7 Either a thermometer or a sensor for temperature measurement to make corrections for pH measurement or an automatic compensation device shall be in use.

5E.5.8 Either a magnetic, TFE-coated stirring bar or a mechanical bar with inert plastic-coated impeller shall be available.

5E.5.9 Records shall be kept showing daily, or before each use, calibration, whichever is less frequent. Calibration shall be performed with at least two buffers in the pH range expected in the samples.

5E.5.10 Aliquots of standard pH 4 & pH 7, or pH 7 & pH 10 shall be used only once.

For conductivity meter:

5E.5.11 A conductivity meter with an error not exceeding 1% or 1  $\mu$ mhos/cm, whichever is greater, shall be in use.

5E.5.12 Records shall be kept to show a daily, or before each use, verification check, whichever is less frequent.

5E.5.13 Records shall be kept showing that the cell constant is determined annually.

For glassware:

5E.5.14 Glassware shall be cleaned in a manner appropriate for the analytical procedures for which it is used including protocols for: metals, ammonia, phosphorus, volatiles and semivolatiles. These cleaning procedures shall be documented.

For refrigerators:

5E.5.15 The bulb of the thermometer in each refrigerator shall be immersed in liquid.

5E.5.16 Thermometers shall be graduated in increments no larger than 1°C.

5E.5.17 Records shall be kept to show that refrigerator temperatures are maintained in the range of 2 - 5°C  $\pm$  1°C.

5E.5.18 Samples to be analyzed for volatile organic compounds (VOCs) shall be stored in separate refrigerators from all other samples.

For ovens:

5E.5.19 Thermometers shall be graduated in increments no larger than 1°C.

5E.5.20 If oven temperature cannot be read without opening the door, the bulb of the thermometer shall be immersed in a sand bath. (A second thermometer should be used to evaluate hysteresis of operation.)

5E.5.21 Oven temperatures shall be controlled and monitored (e.g., beginning and end of each use cycle) to meet applicable method requirements.

For microwave ovens:

5E.5.22 The calibration of the power available for heating shall be documented at least weekly in order to determine that the microwave has not started to degrade and that absolute power settings (watts) may be compared from one microwave to another. (EPA 600/8-91/213; NTIS PB92-114172)

For hot plates:

5E.5.23 Monitor temperature at the center of the hot plate where appropriate and document results. (Note: An uncovered beaker containing 50 ml of a liquid such as an oil located in the center of the hot plate can be used to estimate the temperature.)

For incubators/water baths:

5E.5.24 Method specific temperature requirements shall be controlled and monitored during the course of a test and appropriate records maintained to assure compliance.

For thermometers:

5E.5.25 The laboratory shall have access to a NIST-traceable thermometer.

5E.5.26 The calibration (correction factors) of working liquid-in-glass thermometers shall be checked at least annually against a NIST-traceable certified thermometer.

5E.5.27 The calibration (correction factors) of dial-type thermometers shall be checked at least quarterly against a NIST-traceable thermometer.

5E.5.28 The NIST-traceable thermometer(s) shall be calibrated at least every five years.

For autopipetors/dilutors:

5E.5.29 Apparatus having sufficient sensitivity for the application shall be in use.

5E.5.30 Records shall be kept showing delivery volumes are checked gravimetrically, as appropriate, each month of use.

## **5.6 Measurement Traceability**

The laboratory shall:

5E.6.1 Use quality control materials and calibration standards that are traceable to appropriate national/international measurement standards, where available.

5E.6.2 Document the frequency, conditions, and standards used to establish calibration of all analytical/testing methodology.

5E.6.3 Verify and document all working standards versus primary (reference) standards where available.

5E.6.4 Document the traceability of the specific calibration, calibration check, control, or reference

standards, samples, or mixtures of such standards or samples used to establish or verify the validity of the analytical measurement.

5E.6.5 Label reference materials/reagents with concentrations, date of preparation, expiration date, and the identity of the person preparing the reagent.

5E.6.6 Refrain from using any material beyond the specified expiration date unless documented procedures have been followed for assigning a revised expiration date.

5E.6.7 Have standards preparation documentation such as a preparations record book.

5E.6.8 Instrument performance checks shall be carried out before use for analysis of samples. Such checks shall include, as appropriate, evaluation of instrument sensitivity, noise levels and absorbance/emission levels versus historical values. Acceptance criteria shall be stated.

5E.6.9 Standard curves shall be prepared to adequately cover the expected concentration ranges of the samples using a minimum of three data points for each analyte and one blank, unless otherwise specified by the method employed. Acceptance criteria shall be stated. If acceptance criteria are not met, the problem must be resolved before generating reportable data.

5E.6.10 Field testing devices shall be calibrated as required by the testing procedure. Acceptance criteria shall be stated. In the absence of a requirement in the testing procedure, calibration shall be in accordance with the manufacturer's specification. Calibration records shall be maintained.

## **5.7 Sampling**

(No Additions)

## **5.8 Handling of Test Items**

The laboratory shall:

5E.8.1 Have adequate written procedures for receipt, storage, and processing of samples (including, as applicable, trip and field blanks) to ensure that holding times are met;

5E.8.2 Give samples an unambiguous sample identification when logged.

5E.8.3 Maintain a permanent record for sample log-in data.

5E.8.4 Store samples in such a way as to maintain their identity, integrity, stability, and concentration.

5E.8.5 Maintain sample preservation records.

5E.8.6 Follow appropriate documented chain-of-custody procedures, when required.

5E.8.7 Document failure of sample collector to use appropriate containers, preservatives, packaging, and incorrect documentation and labeling upon receipt of samples.

5E.8.8 Samples disposal must meet waste disposal criteria as applicable.

## **5.9 Assuring the Quality of Results**

5E.9.1 Quality Control (QC) Procedures. The laboratory shall comply with the quality control (QC) procedures required by applicable federal or state environmental or public health agencies when testing for specific analytes. The laboratory shall have QC procedures (SOPs) specific to each test technology addressing, as appropriate, the use of:

5E.9.1.1 Reagent/method blank analyses;

5E.9.1.2 Trip blanks and field blanks;

5E.9.1.3 Replicate/duplicate analyses;

5E.9.1.4 Spiked sample analysis;

5E.9.1.5 Blind samples;

5E.9.1.6 Surrogate standards;

5E.9.1.7 Laboratory control samples (LCSs);

5E.9.1.8 Control charts or the equivalent (e.g., quality control database);

5E.9.1.9 Calibration standards, blanks, and calibration devices (e.g., electronic conductivity meter, NIST-traceable thermometer);

5E.9.1.10 Reference material samples; and

5E.9.1.11 Internal standards.

5E.9.2 Quality Control Practices.

5E.9.2.1 The laboratory shall continually evaluate its performance (system process control) for each method and matrix that includes the determination of accuracy and precision.

5E.9.2.2 Supervisory personnel shall conduct a documented review of the data calculations and QC results.

5E.9.2.3 Deviations or deficiencies in QC shall be reported to management and such reports shall be documented. QC data shall be retrievable for all analytical results.

5E.9.2.4 Method detection limits (MDLs) shall be determined and documented.

Note: Confirmation of MDLs shall be done as appropriate or as required by the method.

5E.9.3 Acceptance Limits. Acceptable performance limits for analytical instrumentation and each method shall be documented based upon the continuing statistical evaluation of data generated by the analysis of quality control samples, unless specific minimum acceptance limits are established by the method.

5E.9.4 Where applicable, the following minimum QC shall be practiced in the laboratory:

For Inorganics/ Classical Chemistry:

- 5E.9.4.1 One calibration check standard and associated blank in 10 samples tested; the lab should repeat analysis of all affected samples if the calibration check standard is outside  $\pm 10\%$  of expected value unless the method specifies otherwise (broader acceptance ranges must be fully justified and documented).
- 5E.9.4.2 One reagent, method, or digestion blank (carried through preparation) in 10 (or per batch).
- 5E.9.4.3 One matrix spike in 10 (or per batch).
- 5E.9.4.4 One duplicate or matrix spiked duplicate in 10 (or per batch).
- 5E.9.4.5 One laboratory control sample in 10 (or per batch).

Note 1: Analysis of a low concentration or near MDL (2 to 3 times MDL) standard with each batch of 10 samples or less is recommended to assess analytical system performance near the MDL (required in CLP SOWs for metals when client needs results near MDL).

Note 2: A batch includes up to 10 samples plus the attendant QC samples unless otherwise specified.

#### For Organics:

- 5E.9.4.6 One calibration check standard in 10 samples (or method-specific frequency) inside established control limits; if any are outside the limits, repeat analysis of all affected samples.
- 5E.9.4.7 One reagent, method, or preparation blank (carried through preparation) in 10 (or per batch).
- 5E.9.4.8 One matrix spike in 10 (or per batch).
- 5E.9.4.9 One duplicate or matrix spike duplicate in 10 (or per batch).
- 5E.9.4.10 Internal or external standards and surrogates (where available) shall be used for all samples.
- 5E.9.4.11 As required by the method, one laboratory control sample (consists of a representative matrix spiked with a reference standard containing the target analytes) in 10 (or per batch).

Note: Analysis of a spiked blank water or soil (LCS)(sodium sulfate or silica sand for organics) that is subjected to ALL extractions and cleanups with each batch of 10 samples or less is recommended to evaluate analytical system performance (CLP Statement of Work (SOW) requires analysis of an LCS).

5E.9.5 The laboratory shall establish control limits for all the above types of QC samples and be able to explain and document the basis for such established limits.

5E.9.6 Control Charts. Control charts or control data shall be used to track laboratory performance with the associated acceptance limits for each matrix and to evaluate instrument performance.

### **5.10 Reporting the Results**

(No Additions)

## **PROFICIENCY TESTING (PERFORMANCE EVALUATION) REQUIREMENTS**

Please refer to R103 – General Requirements: Proficiency Testing for ISO/IEC 17025 Laboratories and the associated Annex (R103a).

## APPENDIX A

### ACRONYMS AND GLOSSARY OF TERMS ASSOCIATED WITH THE PROGRAM

#### ACRONYMS

AA	Atomic Absorption or Automated Analysis (wet chemical)
AAS	Atomic Absorption Spectrometry (see FLAA and GFAA)
A2LA	American Association for Laboratory Accreditation
AIHA	American Industrial Hygiene Association
ANSI	American National Standards Institute
AOAC	Int. Association of Official Analytical Chemists International
APHA	American Public Health Association
ASTM	American Society for Testing and Materials
ASTPHLD	Association of State and Territorial Public Health Laboratory Directors
AWWA	American Water Works Association
C	Celsius (temperature unit)
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation & Liability Act
CDC	Centers for Disease Control
CFR	Code of Federal Regulations
CNAEL	Committee on National Accreditation of Environmental Laboratories
CRADA	Cooperative Research and Development Agreement
CLP	Contract Laboratory Program
CRM	Certified Reference Material
EDL	Estimated Detection Limit
ELPAT	Environmental Lead Proficiency Analytical Testing (AIHA/NIOSH)
EMPC	Estimated Maximum (Possible) Concentration used for dioxin and furans analyses
EPA	Environmental Protection Agency (see also USEPA)
EQL	Estimated Quantitation Limit
FIFRA	Federal Insecticide, Fungicide and Rodenticide Act
FLAA	Direct Flame Aspiration Atomic Absorption Spectrometry
GFAA	Graphite Furnace Atomic Absorption Spectrometry
GLP	Good Laboratory Practices (TSCA and FIFRA)
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICB	Initial Calibration Blank
ICP-AES	Inductively Coupled Plasma Atomic Emission Spectrometry
ICP/MS	Inductively Coupled Plasma-Mass Spectrometry
ICV	Initial Calibration Verification
ICS	Interference Check Standard
IDL	Instrument Detection Limit
IEC	International Electrotechnical Commission
IMVL	Interlaboratory Method Validation Study
ISO	International Organization for Standardization
LCS	Laboratory Control Sample
LOQ	Limit of Quantitation
LSA	Laboratory Systems Audit
MBAS	Methylene Blue Active Substances
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MOU	Memorandum of Understanding
NIOSH	National Institute for Occupational Safety and Health

NIST	National Institute of Standards and Technology
NLLAP	National Lead Laboratory Accreditation Program
NTIS	National Technical Information Service
OSW	Office of Solid Waste (USEPA)
PCBs	Polychlorinated biphenyls
PE	Performance Evaluation
PM	Preventive Maintenance
PT	Proficiency Testing
PQL	Practical Quantitation Limit
QA	Quality Assurance
QAMS	Quality Assurance Management Staff
QAPjP	Quality Assurance Project Plan
QAPP	Quality Assurance Program Plan
QC	Quality Control
QM	Quality Manual
RCRA	Resource Conservation and Recovery Act
RE	Relative Error
RPD	Relative Percent Difference
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Re-authorizations Act of 1986
SM	Standard Methods for the Examination of Water and Wastewater
SOP	Standard Operating Procedure
SRM	Standard Reference Material (produced by NIST)
SW	Solid Waste (usually as part of SW846, an EPA RCRA methods document)
TCLP	Toxicity Characteristic Leaching Procedure
TFE	Tetrafluoroethylene (Teflon or equivalent)
TOC	Total Organic Carbon
TOX	Total Organic Halides
TQM	Total Quality Management
TSCA	Toxic Substances Control Act
USEPA	United States Environmental Protection Agency (see also EPA)
WP	Water Pollution
WS	Water Supply
XRF	X-Ray Fluorescence

## GLOSSARY

Accreditation:	A formal recognition that an organization (e.g., laboratory) is competent to carry out specific tasks (e.g., tests) or specific types of tasks. See also <a href="#">Certification</a> .
Acceptance limits:	Data quality limits specified for analytical method performance.
Accuracy:	The closeness of agreement between a test result and an accepted reference value. The term accuracy, when applied to a set of observed values, will be a combination of a random component and of a common systematic error or bias component. Since in routine use, random components and bias components cannot be completely separated, the reported "accuracy" must be interpreted as a combination of these two components. See <a href="#">Precision</a> and <a href="#">Bias</a> .
Aliquot:	See <a href="#">Subsample</a>
Assessment:	The process of obtaining information on an entity to evaluate its conformance or non-conformance with specified criteria. An assessment generally includes or is intended to mean an on-site visit of the entity.

Assessor: An individual who carries out some or all functions related to laboratory assessment (auditor is a common synonym).

Batch: A quantity of material produced or processed in one operation, considered to be a uniform, discrete unit.

Bias: The difference between the population mean of the test results and an accepted reference value. Bias is a systematic error as contrasted with a random error. There may be one or more systematic error components contributing to the bias.

Blank, Analytical: See Blank, Digestion.

Blank, Calibration: See Blank, Initial Calibration and Blank, Continuing Calibration blank.

Blank, Continuing calibration (CCB): A standard solution which contains no analyte and is used to verify blank response and freedom from carryover, analyzed after the CCV and after the Interference Check Standard (ICS).

Blank, Digestion: See Blank, Method.

Blank, Method: A mixture of all reagents used for the extraction or digestion of the matrix to evaluate the process for contamination from the laboratory. This blank will be run through the entire method.

Blank, Field: A sample of media carried to the sampling site, exposed to the sampling conditions (e.g., bottle caps removed), returned to the laboratory, treated as an environmental sample, and carried through all steps of the analysis in order to evaluate possible site contamination sources such as airborne contaminants.

Blank, Laboratory: See Blank, Method.

Blank, Matrix: A sample of the matrix without the analytes of interest that goes through the complete analysis including digestion.

Blank, Initial calibration (ICB): A standard solution that contains no analyte and is used for initial calibration and zeroing instrument response.

Blank, Reagent: See Blank, Method.

Blank, Rinseate: A sample of a "used" cleaning fluid rinse solution, also called an equipment blank (e.g., a final rinse of the device used to collect environmental samples) used in rinsing collection media and equipment prior to use to monitor possible cross contamination.

Blank, Spiked Reagent: A specified amount of Reagent Blank fortified with a known mass of the target analyte(s), usually to determine the recovery efficiency of the method.

Blank, Trip: A sample of media taken from the laboratory to the sampling site and returned to the laboratory unopened, to evaluate the integrity of the sample container and its preparation.

Blind sample: A subsample submitted for analysis with a composition and identity known to the submitter but unknown to the analyst and used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibrate:	To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the calibration standards should bracket the range of planned measurements. See <a href="#">Calibration curve</a> .
Calibration-check:	See <a href="#">Calibration verification</a> .
Calibration-check standard:	See <a href="#">Calibration verification</a> .
Calibration curve:	The graphical relationship between the known values for a series of calibration standards and instrument responses.
Calibration drift:	The difference between the instrument response and a reference value after a period of operation without recalibration. See <a href="#">Continuing calibration verification</a> .
Calibration standard:	A substance or reference material used to calibrate an instrument (often called a calibrant).
Calibration solution:	See <a href="#">Calibration standard</a> .
Calibration verification:	See <a href="#">Initial or continuing calibration verification</a> .
Certification:	A procedure by which a third party gives written assurance that a product, process, or service conforms to specified requirements. See also <a href="#">Accreditation</a> .
Certified Reference Material (CRM):	A reference material that has one or more of its property values established by a technically valid procedure and is accompanied by or traceable to a certificate or other documentation issued by a certifying body. See <a href="#">Certification</a> and <a href="#">Reference material</a> .
Chain of custody:	An unbroken trail of accountability that ensures the physical security of samples, data, and records.
Check sample:	An uncontaminated sample matrix spiked with known amounts of analytes, usually from the same source as the calibration standards, to establish the stability of the analytical system or to assess the performance of all or a portion of the measurement system. See also <a href="#">Quality control sample</a> .
Continuing calibration verification (CCV):	A standard solution (or set of solutions) used to verify freedom of excessive instrumental drift with the concentration near mid-range of linear curve.
Control chart:	A graph of some measurements plotted over time or sequence of sampling, together with control limit(s) and, usually, a central line and warning limit(s).
Control sample:	See <a href="#">Laboratory control sample</a> .
Corrective action:	Action taken to correct a deficiency noted in an assessment. See <a href="#">Deficiency</a> and <a href="#">Assessment</a> .
Deficiency:	A failure to comply with one of the requirements of the program usually noted during an assessment. See <a href="#">Assessment</a> .
Duplicate analyses:	The analyses or measurements of the variable of interest performed identically on duplicate samples.

Duplicate samples: Two samples taken from and representative of the same population and independently carried through all steps of the sampling and analytical procedures in an identical manner. Laboratory duplicates are used to assess variance of sub-sampling and analysis. Field duplicate samples are used to assess variance of the total method including sampling and analysis.

Field sample: A sample collected and delivered to the laboratory for analysis.

Initial calibration verification (ICV): A standard solution (or set of solutions) used to verify calibration standard levels whose concentration of analyte is near mid-range of linear curve and is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards.

Instrument maintenance log: A chronological record of preventive and emergency maintenance performed on an analytical instrument. The logs include record of calls, service technician summaries, records of calibration etc.

Interference check standard (ICS): A standard solution (or set of solutions) used to verify accurate analyte response in the presence of possible interferences from other analytes present in samples.

Internal standard: A standard added to a test portion of a sample in a known amount and carried through the entire demonstration procedure as a reference for calibration and controlling the precision and bias of the applied analytical method.

Intralaboratory precision: A measure of the method/sample specific analytical variation within a laboratory, usually given as the standard deviation estimated from the results of duplicate analyses.

Laboratory control sample (LCS): A reference material with an established concentration obtained from a source independent of the instrument calibration and traceable to an appropriate national/international measurement standard or other certified reference material.

Laboratory sample: An aliquot or sub-sample of a field sample upon which laboratory analyses are made and results gathered.

Lot: A set of samples submitted together for laboratory analysis which can be treated as one or more batches.

Matrix: The component or substrate which contains the analyte(s) of interest.

Matrix spike: An aliquot of sample spiked with a known concentration of target analyte(s) prior to sample preparation and analysis in order to document the bias of a method in a given sample matrix.

Matrix spike duplicate: Intralaboratory split samples spiked with identical concentrations of target analyte(s) prior to sample preparation and analysis in order to document the precision and bias of the method in a given sample matrix.

Method performance: A general term used to document the characteristics of a method. These characteristics usually include method detection limits, linearity, precision, accuracy and bias.

Method detection limit (MDL): The minimum concentration of an analyte that, in a given matrix and with a specific method, can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

- Mobile laboratory: A mobile laboratory is a self-contained, mobile facility that moves under its own power or is conveyed on a trailer, and does not remain at a site for more than two years.
- NLLAP requirements: Requirements specified by the EPA National Lead Laboratory Accreditation Program (NLLAP) in order to be accredited for lead analysis in paint, soil and dust matrices by an EPA-recognized laboratory accreditation organization such as A2LA.
- Precision: The closeness of agreement between test results obtained under prescribed conditions. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms.
- Primary standard: A substance or device with a property or value that is unquestionably accepted (within specified limits) in establishing the value of the same or related property of another substance or device.
- Proficiency testing: Methods of checking laboratory testing performance by means of interlaboratory tests.
- Quality assurance (QA): For laboratories, the activity of providing the evidence needed to establish confidence that laboratory data are of the requisite accuracy.
- Quality control (QC): For laboratories, the process through which a laboratory measures its performance, compares its performance with standards and acts on any differences.
- Quality system: For laboratories, the organization structure, responsibilities, procedures, processes and resources needed to ensure that laboratory services satisfy data requirements.
- Quantitation Limits: The maximum or minimum levels or quantities of a target analyte that can be quantified with the certainty required by the data user.
- Reference material: A material or substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or assigning values to materials.
- Reference standard: See Reference material.
- Relative percent difference: A term defined as:

$$RPD = \frac{|R_1 - R_2|}{R} \times 100$$

where  $|R_1 - R_2|$  represents the absolute difference in two values and  $R$  represents the average of the two values.

- Replicate analyses: The analysis of the quantity (or analyte) of interest performed identically on two or more laboratory subsamples of the same field sample within a short time interval.
- Run: A set of consecutive sample measurements.
- Sample log: The document where sample identification, condition, etc is noted when samples arrive at the laboratory. The log is part of the sample tracking system. See Sample tracking.

Sample tracking: A system of following a sample from receipt at the laboratory, through sample processing and analysis, and to final reporting. The system includes unique numbering or bar coding labels and the use of a sample log.

Secondary standard: A standard whose value is based upon comparison with a primary standard.

Site blank: See Field blank.

Spiked matrix: See Spiked sample.

Spiked sample: A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available, used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Split samples: Two or more representative portions taken from a sample or subsample and analyzed by different analysts or laboratories used to replicate the measurement of the variable(s) of interest.

Standard addition: The procedure of adding known increments of the analyte of interest to a sample to cause increases in detection response to subsequently establish by extrapolation of the plotted responses the level of the analyte of interest present in the original sample.

Standard operating procedure (SOP): A written document that details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing a certain routine or repetitive task.

Standard reference material (SRM): A certified reference material produced by the U.S. National Institute of Standards and Technology and characterized for absolute content independent of analytical method.

Stock solution: A concentrated solution of analyte(s) or reagent(s) prepared and verified by prescribed procedure(s), and used for preparing working standards or standard solutions.

Stratification: The division of a target population into subsets or strata which are internally more homogeneous with respect to the characteristic to be studied than the population as a whole.

Subsample: A representative portion of a sample. A subsample may be taken from any laboratory or a field sample.

Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

Validation: The process of substantiating specified performance criteria.

Working standard: See Secondary standard

### Document Revision History

Date	Description
July 1, 2001	<p>Scope (page 3) modified to add alternative methods for metals and MTBE.</p> <p>General Criteria updated to reference ISO/IEC 17025.</p> <p>Specific Criteria numbering and order changed to match ISO/IEC 17025.</p> <p>Document Revision History section added.</p> <p>No other changes made.</p>
July 28, 2003	<p>Scope (page 4) modified to add Ethanol, Methyl <i>tert</i>-Butyl Ether, <i>tert</i>-Butyl Alcohol, Ethyl <i>tert</i>-Butyl Ether, Diisopropyl Ether, <i>tert</i>-Amyl Methyl Ether, Benzene, Ethylbenzene, Toluene, and Xylenes.</p> <p>Scope (page 4) modified to replace total metals with dissolved metals.</p> <p>Scope (page 4) modified to add an alternative method for TPH(GRO)</p> <p>Certification Requirements (page 4) Delete Supplement to the Scope.</p> <p>5E.5.4 (page 11) Reference the A2LA Policy on Measurement Traceability and delete “by an authorized person and a certificate shall be provided and retained identifying traceability to an appropriate national/international measurement standards body, such as the National Institute of Standards and Technology (NIST)”.</p> <p>5E.5.12 (page 12) Deleted calibration and Add verification.</p> <p>Scope (page 4) Added additional requirements for Methods 6010B, 8021B, 8260B, and for the dissolved metals.</p> <p>Title (pages 1, 2, 3) modified to reflect the program’s new name.</p>
June 14, 2004	<p>Scope (page 5) modified to update method changes for Dissolved Cadmium, Dissolved Chromium, Dissolved Lead, TPH-DRO, and TPH-GRO.</p>
September 13, 2005	<p>Updated to reference ISO 17025:2005</p>
February 17, 2006	<p>Revised the test methods and analytes per a conference call with LeRoy Feusner.</p>
February 20, 2008	<p>Revisions made by Karen Halvorsen, STR Program Manager, to update test methods and analytes (include silica gel cleanup step for DRO and remove ethanol from analyte list), change the program and division names to Storage Tank Program and Solid &amp; Hazardous Waste Division, and make minor technical writing edits.</p>