



Designation: D 5522 – 99a

Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid¹

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1. Scope*

1.1 This specification covers specific criteria for evaluating the technical capabilities of laboratories involved in testing, measuring, inspecting, and calibrating activities related to chemical analysis of earth materials. In this specification, earth materials shall mean soil, rock, and contained fluids. For the sake of brevity, the term “laboratory” is used in this practice to represent all the above.

1.2 This specification addresses the minimum requirements for laboratories that analyze earth materials for metals, volatile organic compounds, semivolatile organic compounds, pesticides, herbicides, PCBs, radionuclides, and various other parameters by miscellaneous wet chemistry techniques.

1.3 This specification presents specific criteria to be used in an evaluation, including restrictions, minimum requirements, and benchmarks of compliance for specific tests or for specific types of tests.

1.4 This specification is meant only for the evaluation of facilities performing chemical analysis of earth materials and is in no way intended to be an absolute guide. It shall not replace specific criteria that exist for test methods or that exist as separate standards. In instances where laboratory evaluation sections are included as part of a test method, or where specific criteria for test methods exist as separate standards, those separate criteria should also be considered.

1.5 Minimum requirements for agencies engaged in the physical testing of soil and rock can be found in Practice D 3740.

1.6 The values stated in SI units are to be regarded as the standard.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

¹ This specification is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.99 on Quality Control.

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2. Referenced Documents

2.1 *ASTM Standards:*

D 3740 Practice for Minimum Requirements for Agencies Engaged In the Testing and/or Inspection of Soil and Rock as Used In Engineering Design and Construction²

2.2 *USEPA Publications:*

SW-846 Test Methods for Evaluating Solid Waste³
Methods for the Examination of Water and Wastewater²

3. Summary of Specification

3.1 This specification covers minimum requirements for the following items:

- 3.1.1 Organization of the laboratory and its affiliates,
- 3.1.2 Personnel,
- 3.1.3 Quality system,
- 3.1.4 Facilities and equipment,
- 3.1.5 Calibration,
- 3.1.6 Test methods and procedures,
- 3.1.7 Records,
- 3.1.8 Test reports, and
- 3.1.9 Standard operating procedures.

3.2 The items listed here as criteria to be reviewed during an evaluation are standard items that the laboratory shall be following. This includes items that shall be available during an assessment and that the laboratory personnel shall be able to show are being completed for each analysis type.

4. Significance and Use

4.1 This specification is meant for use when evaluating laboratories engaged in chemical analysis of earth materials.

4.2 The criteria specified in this specification can be used in the process of accreditation.

5. Organization

5.1 The legal name, address, and telephone number of the laboratory must be available.

5.2 An organization chart that shows the following information must be presented in the quality control manual:

² *Annual Book of ASTM Standards*, Vol 04.08.

³ Available from United States Environmental Protection Agency.

*A Summary of Changes section appears at the end of this standard.



- 5.2.1 Ownership or membership,
- 5.2.2 Names of affiliations,
- 5.2.3 Management structure,
- 5.2.4 Principal officers,
- 5.2.5 Directors,
- 5.2.6 Relevant organizational components, and
- 5.2.7 Principal chemists.

5.3 Conflicts of interest among the various affiliations must be noted in the evaluation. Such conflicts of interest should be avoided by the laboratory.

5.4 External and subcontracted technical services must be named by the laboratory, along with the addresses and contacts. The subcontractors must have undergone an assessment by the laboratory and documentation of the findings must be available. These outside technical services must meet the requirements outlined in this specification for the relevant test procedures or for specific types of tests performed by the subcontractor for the primary laboratory.

5.5 The quality-control manual must contain a description of its facilities and a summary of the scope of operations.

5.6 Key management and supervisory personnel in each relevant operating, support, and service unit in the laboratory's functional organization and the reporting relationships must be identified and a job description for each of these personnel included as part of the quality-control manual. Resumes on each of these individuals must be included. Each of these individuals shall understand the extent of their responsibilities.

5.7 Qualifications, accreditations, and recognition of the laboratory by others shall be presented, along with a copy of the certifications, in the quality-control manual.

6. Personnel

6.1 The quality-control manual shall address the means by which all personnel will be trained. The manual shall also address the means by which the records will be maintained for said training and work experience.

6.2 All personnel must undergo an introduction to the quality-control manual as well as to the test procedures for which they will be responsible. Appropriate documentation of this training shall be available for review. This documentation shall include any reviews along with the date of the review, a listing of in-house training and certifications from outside training courses, and documented evidence of the analyst's proficiency for each test method performed. This documentation shall be available for review upon request. No analyst shall perform a given test procedure without the appropriate training.

6.3 Each person with the following duties or titles must meet or be supervised by a person who meets the specified minimum experience or have an appropriate educational background:

- 6.3.1 Atomic absorption/ICP supervisor, two years,
- 6.3.2 Atomic absorption analysis, one year,
- 6.3.3 Atomic absorption/ICP sample preparation, three months,
- 6.3.4 Gas chromatography supervisor, two years,
- 6.3.5 Gas chromatography analysis, six months,
- 6.3.6 Gas chromatography spectral interpretation, two years,

- 6.3.7 Purge and trap analysis (GC), six months,
- 6.3.8 Extraction and concentration expert, one year,
- 6.3.9 PCB and pesticide residue analysis expert, two years,
- 6.3.10 General chemistry and instrumentation, six months,
- 6.3.11 GC/MS supervisor, two years,
- 6.3.12 GC/MS operator, four months full-time,
- 6.3.13 GC/MS spectral interpretation, two years,
- 6.3.14 Purge and trap analyst (GC/MS), six months,
- 6.3.15 Microbiology supervisor, one year,
- 6.3.16 Radiochemistry supervisor, five years,
- 6.3.17 Radionuclides analyst, one year,
- 6.3.18 Gross alpha/beta technician, six months,
- 6.3.19 Visible spectroscopy supervisor, two years,
- 6.3.20 Visible spectroscopist, one year,
- 6.3.21 Spectral interpretation (visible spectroscopy), two years, and
- 6.3.22 Inorganic sample preparation—3 months.

7. Quality System

7.1 The quality system must be documented in a manual or equivalent.

7.2 The quality manual must be available to all personnel.

7.3 The quality manual shall be updated at least annually, but will always be under revision.

7.4 The quality manual shall contain the following:

- 7.4.1 Organizational charts,
- 7.4.2 Staff duties including responsibilities for quality,
- 7.4.3 Feedback and corrective action program for internal problems,
- 7.4.4 Technical complaint handling procedure,
- 7.4.5 Policy for documenting procedures and analysis methods,
- 7.4.6 Procedure for sample collection and preservation if performed by laboratory personnel,
- 7.4.7 Procedure for sample storage and handling,
- 7.4.8 Quality-control requirements for each type of test,
- 7.4.9 Procurement and inventory procedures,
- 7.4.10 Policy on the operation and calibration of instruments,
- 7.4.11 Policy on preventative maintenance,
- 7.4.12 Procedure for record keeping and record storage,
- 7.4.13 Procedure for checking the reliability of data reduction and reporting,
- 7.4.14 Procedure for correcting erroneous reports, and
- 7.4.15 Record retention policy.

7.5 The quality-assurance (QA) manager shall have direct access to top management and operate independently of the rest of the laboratory.

7.6 The QA manager should have the power to oversee the laboratory procedures, identify problems, and make recommendations to management.

7.7 A method shall exist so that any deviations or deficiencies in QC are reported to management and such reports are documented.

7.8 All new employees must be given a copy of the quality manual and be required to read it.

7.9 All employees must be given a copy of any changes or additions to the quality manual and be required to review the manual at least once per year.



7.10 All personnel must be trained or prove proficiency for any test method they perform. The training can either be provided in-house or by a certified training school.

7.11 At least quarterly, the QA manager or designee shall conduct an internal audit of the laboratory and report his findings to the laboratory director.

7.11.1 Any problems discovered during the quarterly audit shall be corrected and the steps taken to correct the problem documented.

7.12 All supervisory staff should be aware of the QA/QC system and its application to the daily activities of the laboratory.

7.13 Standard curves that adequately cover the expected sample concentration ranges shall be prepared at least annually or more often as required by the specific test methods. A new curve shall also be prepared when new reagents are prepared.

7.14 Standard curves shall be prepared with a minimum of three standards and one blank or as specified by the method.

7.15 A procedure shall exist such that records indicate what corrective action has taken place when analytical results fail to meet QC criteria.

7.16 Supervisory personnel shall review the data calculations and all QC results.

7.17 The QC data shall be retrievable for all analytical results.

7.18 The method detection limits for all analyses shall be determined and the results documented.

7.19 Computer software programs shall be documented and validated.

7.20 All clients shall be informed if their work is subcontracted and to whom the work is subcontracted.

7.21 All subcontract laboratories shall be evaluated for QA following the guidelines outlined in this practice.

7.22 The laboratory shall perform routine analyses of solvents used for dilutions and extractions to check for contamination.

7.23 The laboratory shall analyze trip blanks as requested by the client or when necessary as indicated by associated samples.

7.24 Chain-of-custody records shall be maintained for all samples and shall be reported with the data when requested.

7.25 The laboratory shall analyze either field duplicates or laboratory duplicates with every group of 20 samples or once per analysis run, whichever is most appropriate (when physically possible given the sample type).

7.26 The precision of the duplicate analyses shall be calculated and the results recorded.

7.27 The laboratory shall have a record of whether it has any history of contamination problems and, if so, what has been done to correct it.

7.28 A reagent or method blank shall be analyzed with every run sequence.

7.29 Spiked samples or blank spikes shall be analyzed once every 20 samples or once per analysis day, whichever is most frequent.

7.30 Blind samples for each analytical procedure shall be analyzed at least quarterly if available.

7.31 Surrogate standards shall be added to all organic samples and organic QC samples whenever possible.

7.32 Blind quality-control samples shall be analyzed at least quarterly by each analyst who performs a given test.

7.33 Training records shall be maintained for all analysts.

7.34 Calibration procedures shall be documented for all test procedures and shall be available to the appropriate personnel.

7.35 Reference standards shall be available and in use as needed. They shall whenever possible be traceable to the National Institute of Standards and Technology (NIST or NBS).

7.36 Quality control check standards shall be analyzed at least once every ten samples with a minimum of one per batch and shall be within the set limits. If the limits are exceeded, all samples analyzed since that last QC standard must be reanalyzed.

7.37 Standard operating procedures (SOPs) shall be available for all test methods and all QC procedures and policies.

7.38 All test samples must be identified with unique identification numbers.

7.39 Only the supervisor (or his designee) in each of the testing areas or their superior shall have the authority to sign test reports or release test data.

8. Facilities and Equipment

8.1 The laboratory shall be controlled with limited access. Limited access being defined as direct entrance by select laboratory personnel with all other access monitored in an appropriate manner.

8.2 The equipment shall be protected from harmful conditions such as exposure to acid fumes, extreme heat, and excessive dust.

8.3 The laboratory environment shall be monitored for proper air flow, ventilation, humidity, and temperature.

8.4 There shall be adequate work space so that each test procedure can be performed safely and efficiently with the least possibility for cross contamination.

8.5 The lighting shall be such that all tests can be performed adequately. For example, titrimetric color changes are easily noted.

8.6 Stable power supplies shall be available. Power regulators shall be used for all major pieces of analytical equipment such as the GC/MS.

8.7 There shall be a source of distilled/demineralized water that has been demonstrated to be free of interferences and contaminants at the necessary detection limits.

8.8 The conductivity of the distilled/demineralized water supply shall be checked daily with the result recorded and shall meet the specifications of the system manufacturer.

8.9 Sufficient exhaust hoods shall be available for volatile/hazardous materials. The hood flow shall be checked at least annually, with a recommendation of every six months.

8.10 Contamination-free work areas shall be present for low-level analyses and microbiological testing.

8.11 Proper work areas, shall be present for handling hazardous chemicals with adequate protection in the case of spillage. This may include the use of stainless-steel trays, plastic trays, or absorbent material.

