Designation: D 5522 – 99a

Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid[1]

This standard is issued under the fixed designation D 5522; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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.*

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 Construction[2]

2.2 *USEPA Publications:*

SW-846 Test Methods for Evaluating Solid Waste[3]

Methods for the Examination of Water and Wastewater[2]

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:

[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] *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.

8.12 Separate cold storage areas shall be available for volatile samples, extracts, standards, reference materials, and other samples as outlined by EPA preservation criteria. The storage areas shall be maintained at 4 ± 2°C and be kept secured/controlled.

8.13 Adequate procedures and facilities shall be available for the collection, storage, and disposal of chemical wastes.

8.14 Proper storage facilities, which are in use, shall be available for volatile, corrosive, explosive, and flammable materials.

8.15 Testing procedures shall be adequately separated to avoid possible cross contamination due to vapors, aerosols, dust, etc.

9. General Test Equipment Requirements

9.1 Appropriate and up-to-date instrument operating manuals and SOPs shall be made available to the analysts. The manufacturer's recommendations and procedures shall be used unless superseded by the specific test method.

9.2 Analytical balances shall be capable of measuring to meet the requirements of the test procedure.

9.3 The area around the balances shall be appropriately cleaned and free from drafts.

9.4 Evaporation and filtration equipment shall be well cleaned.

9.5 The desiccator and the desiccant shall be in good condition.

9.6 The drying ovens shall be electrically safe and capable of reaching and maintaining the required temperatures. If the temperature of the oven cannot be read without opening the oven door, the thermometer bulb shall be immersed in sand.

9.7 Muffle furnace temperatures shall be achievable as required.

9.8 The pH meter shall have the appropriate electrode with scale graduations of at least 0.1 pH units. A temperature sensor for automatic calibration shall be used or a thermometer for manual corrections shall be in place. The probe shall be stored in accordance with the manufacturer's recommendations when not in use.

9.8.1 The buffer solutions shall not exceed the manufacturer's labeled expiration date and be stored in a polyethylene bottle. The aliquot of used buffer solutions shall be discarded after each days use.

9.9 A magnetic stirrer with a PFTE-stir bar shall be available.

9.10 A conductivity meter and a probe of sufficient sensitivity shall be in use.

9.11 Appropriate glassware shall be available and only Class A glassware used for volumetric measurements. All Class A glassware shall be segregated from all other glassware.

9.12 All refrigerators shall be capable of maintaining the required temperature and shall be monitored.

9.12.1 The thermometer bulb shall be immersed in liquid and the thermometers shall have increments no larger than 1°C.

9.13 *Atomic Absorption Spectrophotometer (AA)*:

9.13.1 The AA shall have a grating, a photomultiplier detector, and adjustable slits. The AA shall also be capable of being adjusting between 190 and 800 nm.

9.13.2 The fuels and oxidants are to be of commercial grade or better.

9.13.3 A filter moisture trap shall be in use between the air source and the spectrophotometer.

9.13.4 The nitrous oxide shall be of reagent grade or better.

9.13.5 Backflash arresters and heaters shall be in place where needed.

9.13.6 Burner head gases shall be removed by ventilation.

9.13.7 All gages and couplings shall be correctly mated.

9.13.8 Proper burner heads shall be available and in use. They will be clean and free of buildup.

9.13.9 For graphite furnace AAs, the tube shall be changed at least every six months or as needed and the chamber cleaned. Performance of this procedure shall be documented in the maintenance log.

9.13.10 Single-element lamps are preferred, but not required. The date when each is first put into use shall be noted.

9.13.11 Background correction capabilities shall be available.

9.13.12 If a cold vapor mercury analyzer or attachment is used, an absorption cell with quartz windows on each end shall be available. In addition, any other equipment required by the method shall be available.

9.14 *Inductively Coupled Plasma (ICP)*:

9.14.1 Background correction shall be available and in use for the ICP.

9.14.2 The nebulizer shall be free of salt buildup and the method used to control this shall be noted. Rinsing with a method blank between samples is the preferred method.

9.14.3 The ICP shall be equipped with an argon gas supply.

9.14.4 Whether the ICP is a sequential or simultaneous element analyzer shall be noted.

9.15 *Visible Spectrophotometer*:

9.15.1 The cell compartment of the spectrophotometer shall be able to accommodate the cell sizes which are needed to perform the specific task.

9.15.2 The cells shall be clean and free of scratches, finger prints, and evaporated film residue.

9.15.3 The lab shall possess at least one pair of matched cells with documented equivalency checks.

9.15.4 The spectrophotometer shall be capable of reading to wavelengths needed to perform the test of interest.

9.15.5 For an automated spectrophotometer, there shall be a chemical drain in place and the following items in use: a sampler, continuous filter, proportioning pumps, analytical cartridges as required, manifolds as required, colorimeter with various filters and flow cells, recorder, heating baths as required, a block digester, and a digital printer. All tubing diameters shall be appropriate for the analyses.

9.16 *Automatic Titrators*—Automatic titrators shall be used in accordance with the manufacturer's instructions and shall be properly maintained.

9.17 *Electronic Probes*:

9.17.1 The meter used shall either have an expanded millivolt scale or read directly in concentration units.

9.17.2 Each electrode used shall be appropriate to the test procedure.

9.17.3 A sleeve-type (non-fiber) or combination electrode shall be available.

9.17.4 The analyst must understand potential interferences for the probes in use.

9.17.5 The meter shall be allowed to warm up for the time recommended by the manufacturer.

9.17.6 For the dissolved oxygen electrode, the membrane shall be changed frequently and the appropriate membranes and electrolyte available.

9.18 *Gas Chromatograph (GC)*:

9.18.1 The GC oven shall be capable of temperature control within ±1.0°C up to a temperature of 300°C.

9.18.2 The following detectors shall be available when necessary and shall be used properly:

9.18.2.1 Electrolytic conductivity,

9.18.2.2 Microcoulometric,

9.18.2.3 Photoionization,

9.18.2.4 Flame ionization,

9.18.2.5 Electron capture,

9.18.2.6 Nitrogen/phosphorus,

9.18.2.7 Flame photometric,

9.18.2.8 Hall, and

9.18.2.9 Thermal-energy analyzer.

9.18.3 If a chart recorder is in use, it shall have a chart width of at least 10 in. (254 mm), be able to give a full-scale response in no more than 1 s, have a signal that matches the instrument, and shall have an adjustable chart speed.

9.18.4 The purge-and-trap system shall be capable of providing finely divided gas bubbles throughout the sample by means of the purge inlet gas device. It shall also be capable of retaining compounds at room temperature.

9.18.5 The desorber for the purge-and-trap unit shall be capable of heating the trapping device to at least 180°C with less than 40°C overshoot.

9.18.6 The purge-and-trap unit shall be capable of accepting 5-mL samples with a gaseous headspace of less than 15 mL.

9.18.7 The trap shall have a length of at least 20 cm.

9.18.8 Appropriate columns for primary and confirmation runs shall be available for the various test parameters.

9.19 *Gas Chromatography/Mass Spectroscopy (GC/MS)*:

9.19.1 It is preferable that all GC/MSs be programmable.

9.19.2 The GC/MS interface shall be glass or glass lined and a split/splitless capillary injection system shall be in place.

9.19.3 The MS shall be capable of scanning from 70 to 450 mass units every 7 s or faster.

9.19.4 The computer system shall be capable of collecting data continuously throughout the entire chromatographic run.

9.19.5 The computer system software should contain the most recent spectral library available.

9.19.6 The computer software shall allow integrating the abundance in any extracted ion current profile between specified time or scan number limits.

9.19.7 In-house replacement parts shall be available for those items that are consumable and often replaced.

9.19.8 Appropriate GC columns shall be available for the specific analysis methods.

9.20 *High-Performance Liquid Chromatography (HPLC)*:

9.20.1 The proper columns and syringes shall be in place.

9.20.2 The HPLC shall contain the detector most appropriate for the compounds of interest.

9.20.3 Some type of recorder or integrator plus a data reduction system shall be in place.

9.20.4 The HPLC system shall have the appropriate injection system.

9.20.5 The laboratory shall possess an electrode polishing kit.

9.20.6 Consistent volume injection loops shall be in use or a system to record the injected volume to the nearest 0.5 µL.

9.20.7 The mobile phase shall be prepared at least weekly and degassed daily.

9.21 *Ion Chromatography (IC)*:

9.21.1 The IC shall contain an anion-guard column, an anion-separator column, an anion-suppressor column, a conductivity cell detector, and either a strip-chart recorder or an integrator.

9.21.2 Particle sizes larger than 0.20 µm shall be filtered from both samples and solutions.

9.21.3 The reagent water shall be free of the anions of interest.

9.21.4 Nitrite and phosphate working standards shall be prepared daily and all other working standards prepared on at least a weekly basis.

9.21.5 The same size sample loop shall be used for both the samples and the standards.

9.22 *Heating Blocks*—Heating blocks shall be capable of achieving and maintaining a temperature of at least 150 ± 2°C.

9.23 *Total Organic Carbon (TOC) Analyzer*:

9.23.1 *Combustion–Infrared*—A means shall exist for the reduction of particle size since sample introduction requires a small particle size. There shall be a separate chamber for the measurement of inorganic carbon. All contact with organic matter shall be avoided before and during the analysis. The carrier gas shall be CO_2 free and contain less then 1 ppm hydrocarbon. The instrument shall be stabilized at 900°C before use and a homogenized blank run before any samples. The syringe size shall be compatible with the particle size in the sample. (IC correction)

9.23.2 *Persulfate–Ultraviolet Oxidation*—The TOC analyzer shall have a nondispersive infrared analyzer along with a flame ionization detector and a chemical titrator. Particle size reduction shall be performed when necessary and glass fiber filters shall be acid washed before use.

9.23.3 *Wet Oxidation*—This method is only applicable for low-level nonpurgeable organic carbon. The potassium persulfate shall be granular and the glass fiber filters acid washed.

9.24 *Total Organic Halide (TOX) Analyzer*—All glassware shall be cleaned with an adequate cleaning solution and muffle furnace fired at 400°C (except volumetric glassware) for 15 to 30 min. The purity of the activated carbon shall be verified before use and the adsorption efficiency checked. The pyrolysis of the sample shall be done in an oxygen-rich environment and the possibility of breakthrough on heavily contaminated samples checked.

9.25 *Pensky Martin Closed Cup Flash Tester*:

9.25.1 The instrument shall not be modified in any way.

9.25.2 Two thermometers shall be present.

9.25.3 Heating of the samples shall be done with care and at a rate that will avoid the loss of sample.

9.25.4 The flash point shall be adjusted for barometric pressure.

9.26 *Setaflash Closed Cup Flash Tester*:

9.26.1 Heat transfer tape shall be in use and the test apparatus placed in subdued light and out of disturbing drafts.

9.26.2 A magnifying glass shall be available to read the thermometer.

9.27 *Extraction Procedure (EP) Toxicity Test Apparatus*:

9.27.1 The extractor shall be built in accordance with the suggested design and allow for mixing such that stratification of the sample does not occur.

9.27.2 The extractor shall be maintained such that the sample is rotated at a rate of 20 rpm.

9.28 *Toxic Characteristic Leaching Procedure (TCLP) Apparatus*:

9.28.1 *Agitation Apparatus*—The extraction vessel shall rotate end-over-end at a rate of 30 ± 2 rpm with the temperature of the area monitored and recorded.

9.28.2 *Extraction Vessel (TCLP)*—The extraction vessel shall be made of plastic-coated borosilicate glass and have a volume of 2 L.

9.28.3 *Extraction Vessel (Zero Head Extractions (ZHE))*—The ZHE shall be a commercial device that is pressure checked for leaks after each use. The vessels shall be made of either glass, PTFE, or 316 stainless steel. HDPE, PVC, or polypropylene devices are to be used only for metals mobility tests.

9.29 *Autoclaves*:

9.29.1 The autoclave shall be capable of reaching a sterilization temperature of 121°C, maintain that temperature for no more than 15 min, and require no more than 45 min for a complete cycle.

9.29.2 Temperature and pressure gages shall be on the exhaust side on a flow-through autoclave along with an operating safety valve.

9.29.3 The autoclave shall depressurize at a slow enough rate so that the culture media do not boil over.

9.30 *Ultraviolet (U.V.) Sterilizer*—The sterilizer shall be properly disinfected before each use and the bulb replacements done on a routine basis.

9.31 *Incubators*:

9.31.1 The incubators shall be large enough to prevent overcrowding of the samples and have an internal temperature-monitoring device sensitive to ±0.5°C.

9.31.2 The temperature shall be maintained at the appropriate temperature for the microbiological test being performed.

9.32 *Water Baths*:

9.32.1 The water baths shall be large enough to prevent overcrowding of samples.

9.32.2 Circulating water baths are recommended for all intended uses but are only required when microbiology samples are being prepared. Any other water bath such as for mercury digestions may be noncirculating.

9.33 *Filtration Equipment*:

9.33.1 The filtration unit shall be made of a material suitable for the test procedure and be used with the appropriate size and type of filter.

9.33.2 If pressure filtration is used, the pressure shall never exceed 50 psi.

9.34 *Radiochemistry Equipment*:

9.34.1 All detectors shall be stored in graded lead shielding.

9.34.2 The instruments shall be in a room separate from where samples and standards are handled and prepared for analysis.

10. Reagents

10.1 All chemicals and reagents shall be labeled, dated, and signed with the date of receipt or the day the reagent is prepared.

10.2 All chemicals and reagents shall be proven free of contaminants and interferences.

10.3 All acids shall be of reagent grade or better, except for those used for ICP work, which need to be of high-purity grade or equivalent.

10.4 All solvents shall be of chromatographic grade or better.

10.5 A log book shall be maintained of all reagents.

11. Calibration

11.1 A program shall exist for initial and periodic calibration of all equipment such that the frequency, conditions, standards, and calibration history are documented.

11.2 All reference material and where applicable, all measurements shall be traceable to an appropriate agency, this includes calibration standards. Document the preparation. The documentation shall include the solvent, concentration, date, preparer's name, and the expiration date.

11.3 Use only primary reference standards for calibration.

11.4 Verify all working standard concentrations versus the primary standard and document the comparison.

11.5 Calibration protocols for all the analytical instrumentation shall be available to the analysts.

11.6 Keep all calibration results in a permanent record.

11.7 Whenever possible, use a quality-control sample to verify calibration standards.

11.8 Keep maintenance logs on each piece of analytical equipment and recalibrate all equipment following any type of repair or when the performance of the equipment is in doubt.

11.9 *Analytical Balances*:

11.9.1 Check each analytical balance daily (or with use) with a minimum of one Class S or S-1 weight in the range in which the measurements will be made. Check the balance monthly with a series of Class S or S-1 weights and document the results. Any variance of greater than 0.1 % between the expected weight and the actual weight requires corrective action.

11.9.2 At least annually, calibrate the balance by a certified technician and document the calibration.

11.10 *Class S Weights*—Calibrate the Class S weights that have been calibrated within the last five years and are traceable to NIST (NBS).

11.11 *Drying Ovens*—Check the temperature of each oven before and after each usage to verify the correct operating temperature for the given test procedure. Document all checks in a bound logbook.