Designation: D7448 – 16

Standard Practice for Establishing the Competence of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke[1]

This standard is issued under the fixed designation D7448; 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 practice specifies requirements to operate and evaluate the quality and management systems in a laboratory that provides services with respect to sample collection, sample preparation, or testing of coal, coke, and ash derived from coal or coke using ASTM standards that are under the jurisdiction of Committee D05 on Coal and Coke.

NOTE 1—The word "laboratory" is used throughout this practice when referring to an organization that provides services in coal sampling or testing, or both. It is recognized, however, that the word may not be appropriate to an organization that does not perform actual laboratory sample testing.

1.2 International standard ANSI/ISO/IEC 17025 shall be the governing document specifying requirements for management, technical competence and evaluation of a laboratory.

NOTE 2—An accrediting body or user of laboratory services can also impose technical or non-technical requirements not specifically addressed in ANSI/ISO/IEC 17025 provided they do not invalidate the requirements of ANSI/ISO/IEC 17025.

1.3 This practice is used to evaluate only those capabilities specifically claimed by a laboratory.

1.4 All percentages are percent mass fractions unless otherwise noted.

1.5 *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:*[2]

D121 Terminology of Coal and Coke

D3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal

D4239 Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High-Temperature Tube Furnace Combustion

D4749 Test Method for Performing the Sieve Analysis of Coal and Designating Coal Size

D5061 Test Method for Microscopical Determination of the Textural Components of Metallurgical Coke

D5515 Test Method for Determination of the Swelling Properties of Bituminous Coal Using a Dilatometer

D6172 Test Method for Determining the Volume of Bulk Materials Using Contours or Cross Sections Created by Direct Operator Compilation Using Photogrammetric Procedures

E2554 Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques

2.2 *ANSI/ISO/IEC Document:*[3]

17025 General Requirements for the Competence of Calibration and Testing Laboratories

2.3 *ISO Documents:*[4]

5725-3 Accuracy (Trueness and Precision) of Measurement Methods and Results—Part 3: Intermediate Measures of Precision of a Standard Measurement Method

5725-6 Accuracy (Trueness and Precision) of Measurement Methods and Results—Part 6: Use in Practice of Accuracy Values

3. Significance and Use

3.1 International standard ANSI/ISO/IEC 17025 promotes the use of documented accountability and quality control procedures to assure a laboratory and its clients that the laboratory can produce technically valid data and results in the routine performance of its sampling, sample preparation and testing activities.

3.2 A laboratory shall use ANSI/ISO/IEC 17025 to develop its quality system. Clause 4 of ANSI/ISO/IEC 17025 specifies the requirements for sound management. Clause 5 of ANSI/

[1] This practice is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.24 on Statistics.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[4] Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

ISO/IEC 17025 specifies the requirements for technical competence for the type of tests or calibrations, or both the laboratory undertakes.

3.3 In addition to complying with the requirements of ANSI/ISO/IEC 17025, the Annex of this standard practice contains information that shall be considered important for the evaluation and operation of a competent Coal and Coke sampling or testing facility, or both. The information in this Annex is presented where it is not otherwise covered in ANSI/ISO/IEC 17025 or the applicable ASTM methods.

3.4 Laboratory clients, regulatory authorities, and accreditation bodies that recognize the competence of testing and calibration laboratories can use this standard practice as the basis for their evaluation.

3.5 The primary significance of this practice is to establish that for a laboratory to generate measurements traceable to SI units, it must:

3.5.1 Have a clear understanding of the work requested by the client.

3.5.2 Meet the quality system requirements of the internationally accepted ANSI/ISO/IEC 17025 standard.

3.5.3 Use test methods which have been shown to be traceable to SI units of measurement.

3.5.4 Be able to demonstrate that the laboratory is in statistical control at the time the measurements are made.

4. Selection of an Evaluator

4.1 The evaluator(s) shall have sufficient technical background to competently evaluate the application of ASTM standards employed for the sampling, preparation and analysis of coal, coke, and ash derived from coal or coke.

4.2 The evaluator(s) shall review the laboratory with the aid of a worksheet or checklist. Worksheets or checklists shall be developed from the requirements of Clause 4 and Clause 5 of ANSI/ISO/IEC 17025 in concert with specific technical requirements specified in the ASTM standard(s) to be evaluated.

4.3 Observations and comments made by the evaluator shall be keyed to item numbers in the checklist.

5. Keywords

5.1 accuracy; competence; evaluation; laboratory; management; stability

ANNEX

(Mandatory Information)

A1. FACTORS AFFECTING THE TECHNICAL VALIDITY OF TESTS

INTRODUCTION

Factors that can influence the technical validity of tests performed by a laboratory include but are not limited to:

(1) Sampling apparatus operation,
(2) Sampling procedures,
(3) Sample preparation apparatus,
(4) Sample preparation operation,
(5) Sample preparation procedures,
(6) Laboratory equipment,
(7) Laboratory equipment operation,
(8) Test methods,
(9) Calibration, control and consumable materials,
(10) Calibration,
(11) Uncertainty of measurement,
(12) Control charts,
(13) Reporting procedures,
(14) Qualification of laboratory personnel, and
(15) Environmental conditions (sampling, sample preparation and analyses).

This annex while mandatory, identifies several items as a “typical practice”. A “typical practice” is not to be considered the only correct practice. Typical practices have been included throughout this Annex only for the purpose of clarifying the content of this Annex.

The extent to which the factors contribute to the technical validity of results can differ considerably between tests. The laboratory shall have procedures that address these factors where these are applicable and under the laboratory’s control.

ANSI/ISO/IEC 17025 Clause 4.1.5 states:

“The laboratory shall have managerial and technical personnel who, irrespective of other responsibilities, have the authority and resources needed to carry out their duties, including the

implementation, maintenance and improvement of the management system, and to identify the occurrence of departures from the management system or from the procedures for performing tests and/or calibrations and to initiate actions to prevent or minimize such departures."

Managerial and technical personnel shall have the authority and resources to implement the practices specified in this Annex in a manner that ensures laboratory procedures produce data of the type and quality needed for the intended end use.

A1.1 Sampling Apparatus Operation

A1.1.1 The laboratory shall have procedures describing acceptance requirements for all sampling equipment.

A1.1.2 The laboratory shall have procedures describing verification of all sampling equipment.

A1.2 Sampling Procedures

A1.2.1 The laboratory shall have sampling procedures describing all processes under its control.

A1.2.2 The laboratory shall have procedures describing requirements for bias tests.

A1.2.3 The laboratory shall have procedures describing requirements for periodic bias tests after initial bias testing and acceptance of sampling systems.

A1.2.4 The laboratory shall have procedures describing requirements for bias tests subsequent to maintenance and or/modification of sampling systems.

A1.3 Sample Preparation Apparatus

A1.3.1 The laboratory shall have procedures describing:

A1.3.1.1 Acceptance requirements for all sample preparation equipment.

A1.3.1.2 Procedures for verification of all sample preparation equipment.

A1.4 Sample Preparation Operation

A1.4.1 The laboratory shall have a documented management plan which shall include requirements for:

A1.4.1.1 Maintaining a chain of custody for samples.

A1.4.1.2 Identifying the source of the sample.

A1.4.1.3 Identifying date and time of initial sampling.

A1.4.1.4 Identifying the individual(s) responsible for sampling and sample preparation.

A1.4.1.5 Describing test and reserve sample(s) collected at each stage of sampling and sample preparation including date, time, top size and mass.

A1.4.1.6 Minimizing contamination at each stage of sampling and sample preparation.

A1.4.1.7 Minimizing dust loss and verifying material recovery at each stage of sampling and sample preparation.

A1.4.1.8 Verifying particle size reduction equipment produce samples of the appropriate particle size criteria and homogeneity.

A1.4.1.9 Minimizing changes in moisture and or oxidation of the sample(s) during transportation, preparation, storage and handling.

A1.4.1.10 Mixing to minimize selection of a biased size fraction at each stage of sampling, including extraction of test portions from the laboratory analysis sample.

A1.4.1.11 Recording date and time the sample(s) are received in the laboratory.

A1.4.1.12 Verifying compliance or corrective action with respect to all elements of the management plan, or both.

A1.4.1.13 Approval of changes or exceptions to the sampling and sample preparation procedures

A1.5 Sample Preparation Procedures

A1.5.1 The laboratory shall have procedures for all stages of sample preparation that are under its control.

A1.6 Laboratory Equipment

A1.6.1 The laboratory shall utilize equipment that meets the specifications of the relevant sampling/test methods.

A1.7 Laboratory Equipment Operation

A1.7.1 The laboratory shall have procedures describing equipment set-up and startup where appropriate.

A1.7.2 Equipment that is operated outside of normal parameters shall be subject to verification.

A1.7.3 The laboratory shall have procedures describing the minimum quality control that is required to validate the equipment is in statistical control at the time the measurements are made.

A1.7.4 The laboratory shall maintain all original instrument observations in accordance with their records control procedures.

A1.8 Test Methods

A1.8.1 Laboratory procedures can consist of standard and non-standard methods. A non-standard method includes the application of a standard method to samples that fall outside the scope or validated range of the standard method or that allow conditions of test which depart from those specified in a standard method.

A1.8.2 Publications from the AOAC International,[5] as well as ISO 5725-3 and ISO 5725-6 describe procedures for establishing the precision and accuracy of a non-standard method or demonstrating the equivalency of a non-standard method with a standard method.

A1.8.3 The laboratory shall identify by ASTM (or other source) designation including revision, all standard and non-standard sampling, sample preparation and test procedures employed by the laboratory. The ASTM designation system allows users to determine the potential for technical discrepancies to exist between laboratories claiming to employ the same ASTM test method.

[5] Grant T. Wernimont, *Use of Statistics to Develop and Evaluate Analytical Methods*, Association of Official Analytical Chemists, 1985.

A1.8.4 Table A1.1 provides examples of how this system operates.

NOTE A1.1—Referenced documents within a method or practice always refer to the most current version of the referenced document, even if the referenced document is revised after the ASTM test method or practice is published. In this example, D5061-92 and D5061-92 (Reapproved 2007) both refer to the most current version of the referenced document Terminology D121. Minor text changes can exist due to ASTM technical requirements or grammatical corrections; however, these would not be of a technical nature.

A1.9 Calibration, Control and Consumable Materials

A1.9.1 All calibration, control and consumable materials can degrade.

A1.9.2 Store these materials in a manner that minimizes opportunities for moisture changes, oxidation, or other degradation to occur.

A1.9.3 The laboratory shall have procedures describing practices for receipt, preparation, acceptance, use, storage, expiration and disposal of these materials.

A1.9.4 The laboratory shall have procedures describing requirements for approval of changes or exceptions to selection, handling, storage and use of these materials.

A1.9.5 The laboratory shall have procedures describing practices to ensure calibration, control and consumable materials that leave the jurisdiction of the laboratory are handled appropriately to maintain tractability or integrity, or both. where appropriate these procedures shall include verification by the receiving laboratory.

A1.9.6 The laboratory shall have procedures describing practices to ensure that calibration and control materials that have expired are clearly identified and are not subsequently employed for calibration or verification purposes. These procedures shall ensure that discarded materials can not be subsequently used.

A1.9.7 *Calibration and Control Materials:*

A1.9.7.1 All reference materials must meet the requirements of a certified reference material (CRM) and can include pure substances, pure mixtures, external reference materials (ERMs) and internal reference materials (IRMs) in a solid, liquid, or gaseous state.

A1.9.7.2 Certified reference materials include primary reference materials, secondary reference materials and other reference materials.

(1) A primary reference material is defined by ISO as a reference material "whose quantity value and measurement uncertainty are established without relation to another measurement standard for a quantity of the same kind."[6]

(2) A secondary reference material is defined by ISO as a reference material "whose quantity value and measurement uncertainty are assigned through calibration against, or comparison with, a primary measurement standard for a quantity of the same kind."[6]

(3) An internal reference material is one generated by a laboratory solely for its own use.

(a) The laboratory shall have procedures describing the processes employed to establish calibration and control values that are traceable to reference material(s). Those procedures shall include processes to assign a value and uncertainty and show traceability to a primary reference material where primary reference materials exist.

(4) All reference materials shall meet the minimum requirements of a secondary reference material unless no primary or secondary reference materials are available.

A1.9.7.3 Select reference materials with matrix and values similar to samples routinely tested in the laboratory. Only use a material that has been prepared consistent with the requirements of sampling and sample preparation methods employed for the preparation of routine test samples.

A1.9.7.4 Select control and calibration material(s) to cover the full range of the expected property values and the matrix.

A1.9.7.5 The selection of reference material(s) is to include primary reference material(s) where they are available.

A1.9.7.6 Discard and replace calibration and control materials when less than 10 % of the original mass remains. Before discarding the material conduct tests to verify the acceptability of replacement material.

A1.9.7.7 Prior to use verify the stability of reference materials through expiration dates, control charts or comparison to Primary Reference Materials, or both.

A1.9.7.8 Do not employ the same material(s) for control purposes as employed for calibration or calibration verification purpose(s) where alternate producers or lot numbers of these material(s) exist. The routine testing sequence should include control samples preferably similar in composition to the routine test samples.

A1.9.8 *Consumable Materials:*

A1.9.8.1 Consumable materials shall be selected to meet the requirements of the test and to meet the specifications of equipment used in the conduct of the test.

[6] ISO International Vocabulary of Basic and General Terms in Metrology (VIM): 1993

TABLE A1.1 Using ASTM Standard Designations

Standard Method Designation Lab A	Standard Method Designation Lab B	Technically Equivalent
ASTM D5061-92	ASTM D5061-92 (Reapproved 1997)	Yes: Reapproval indicates no technical changes to standard D5061. (Note A1.1)
ASTM D5515-94	ASTM D5515-97	No: The lab B year designation is 97. The lab A designation is 94. This indicates technical differences between the two methods.
ASTM D6172-98	ASTM D6172-98[ε]	Yes: Epsilon indicates an editorial change added to improve clarity of use.
ASTM D4239-02	ASTM D4239-02a	No: The "a" indicates a technical revision occurred in the same year.

NOTE A1.2—*Example of a Typical Practice*: The laboratory shall have procedures describing procedures for verifying calibration, control and consumable materials are of acceptable homogeneity prior to acceptance and during use. For pure mixtures, inspect liquid solutions for evidence of precipitate or residue. Maintain gas mixtures at temperatures above which phase separation can occur. For heterogeneous solids, such as coal, acceptance can include multiple determinations of a property such as ash content. For coal derived ash acceptance can include multiple analyses of components such as silicon dioxide or iron oxide. Conduct these confirmatory tests concurrent with analysis of a material of acceptable homogeneity such as a certified reference material or reference material traceable to a certified reference material. Include mixing instructions in procedures for subsequent extraction of test portions.

NOTE A1.3—A "typical practice" is not to be considered the only correct practice. Typical practices have been included throughout this Annex only for the purpose of clarifying the content of this Annex.

A1.10 Calibration—Minimum Requirements

A1.10.1 This standard practice describes practices for calibration acceptance and calibration verification.

A1.10.1.1 Calibration acceptance is the process to include or exclude a calibration point in the determination of the regression coefficients.

A1.10.1.2 Calibration verification is the process to confirm the stability of a regression.

A1.10.1.3 To minimize the probability of suspect test results, a test method that specifies calibration employing either pure substances, certified reference materials, or reference materials requires both calibration acceptance and calibration verification.

A1.10.2 A calibration point is a measurement included in the calculation of the regression coefficients.

A1.10.3 Employ only those materials for calibration or calibration verification that specify an expanded uncertainty for the assigned value. This expanded uncertainty is commonly referred to as the confidence interval of the assigned value. Do not employ the same material(s) for calibration verification as employed for calibration purpose(s) where alternate producers or lot numbers of these material(s) exist. Select calibration verification material(s) within the concentration range of the calibration materials.

A1.10.3.1 The expanded uncertainty of the assigned value of any material employed for calibration or calibration verification shall meet conditions of purity specified in the method and shall not exceed the verification criteria.

A1.10.4 A minimum of 4 calibration points is required to apply all of these regression fits.

TABLE A1.2 Recommended Number of Calibration Points for Common Regressions

NOTE 1—The recommended number of calibration points allows the process of calibration acceptance to exclude calibration points while still achieving the minimum number of calibration points.

Regression Fit	Minimum	Recommended
Linear	3	6
Quadratic	4	7
Power	4	7

A1.10.5 Cubic fit is excluded from the allowed regression fit. If a cubic fit is required, preference should be given to reducing the calibration range in order to use the listed regression fits.

A1.10.6 *Calibration:*

A1.10.6.1 Select calibrant material(s) to cover the expected parameter range in test samples.

A1.10.6.2 Comply with the acceptance criteria specified in the section of the method describing the procedure for calibration acceptance.

A1.10.6.3 If a method does not specify acceptance criteria, conduct sufficient measurements for each calibrant to produce 5 calibration points. Select a regression fit that yields the minimum root mean square.

NOTE A1.4—The root mean square is expressed as:

$$Syx = \sqrt{\sum (AV - PV)^2/(NCP - NRC)} \qquad (A1.1)$$

where:

Syx = the root mean square,
AV = the actual or known calibration value (concentration or response),
PV = the value predicted from the regression fit (concentration or response),
NCP = the number of calibration points,
NRC = the number of regression coefficients, and
NCP – NRC = the degrees of freedom.

NOTE A1.5—The term "number of regression coefficients" is synonymous with the terms "order of the equation" or the "coefficient parameters" if the intercept coefficient is included. In the below examples the linear equation has 2 coefficients (m_1 and b), the quadratic equation has 3 coefficients (m_1 , m_2 and b), and the power fit has 3 coefficients (m1, n and b).

(1) In the case of a minimum of five calibration points the equation reduces to:

Linear Fit: $y = m_1x + b$ $\quad Syx = \sqrt{\sum (AV - PV)^2/(5 - 2)}$

Quadratic Fit: $y = m_1x^2 + m_2x + b$ $\quad Syx = \sqrt{\sum (AV - PV)^2/(5 - 3)}$

Power Fit: $y = m_1x^n + b$ $\quad Syx = \sqrt{\sum (AV - PV)^2/(5 - 3)}$

A1.10.7 *Calibration Verification:*

A1.10.7.1 Comply with the criteria specified in the section of the method describing the procedure for calibration verification.

A1.10.7.2 If a method does not specify calibration verification criteria or specifies calibration verification criteria that do not comply with the minimum requirements specified in this standard practice apply the following verification protocol.

A1.10.7.3 Select a verification material based on Table A1.3.

A1.10.7.4 The selection of calibration verification material(s) is to include primary reference material(s) where they are available. Employ other reference materials only when primary reference materials are not available.

A1.10.7.5 Conduct analysis of the verification material.

(1) Where not specified in the method, verify the calibration after a maximum of every 10 test samples. If verification

TABLE A1.3 Calibration Verification Criteria Matrix[A]

Calibration Material	Verification Criteria Specified in Method	Calibration Verification Material	Verification Criteria
Pure Substance(s)	Yes	Pure Substance(s)	Acceptance limits as determined from the acceptance criteria specified in the method
Pure Substance(s)	No	Certified Reference Material or Reference Material Traceable to a Certified Reference Material	Measured value agrees with assigned value within the reproducibility specified in the method
Certified Reference Material or Reference Material Traceable to a Certified Reference Material	Yes	Certified Reference Material or Reference Material Traceable to a Certified Reference Material	Measured value agrees with assigned value within the reproducibility specified in the method
Certified Reference Material or Reference Material Traceable to a Certified Reference Material	No	Certified Reference Material or Reference Material Traceable to a Certified Reference Material	Measured value agrees with assigned value within the reproducibility specified in the method

[A] For additional information, see ISO 5725-6 Accuracy (Trueness and Precision) of Measurement Methods and Results-Part 6: Use in Practice of Accuracy Values.

fails, reject all test results back to the last acceptable verification. Take corrective action to identify the assignable cause and recalibrate the instrument.

(2) If the predicted value for the verification material agrees with the assigned value within verification criteria given in Table A1.3, proceed with analysis of the test samples.

(3) If the verification run does not agree within the verification criteria immediately conduct an additional duplicate determination of the verification material. If each duplicate determination agrees with the assigned value within the verification criteria, proceed with analysis of the test samples. Otherwise the calibration is suspect. Do not discard any verification results.

A1.10.8 *Example 1—ASTM Test Methods D4239 Method A:*

A1.10.8.1 A lab employs Test Methods D4239 Method A [Reproducibility limit = 0.02 + 0.09*(average sulfur value)] for the determination of sulfur. The expected range of sulfur concentration is 0.60 to 3.50 %.

A1.10.8.2 Test Methods D4239 Method A specifies the use of certified reference materials, or reference coals for calibration. Test Methods D4239 Method A includes a section on periodic calibration verification. A minimum of three calibration points is required (see Table A1.2).

A1.10.8.3 Since Test Methods D4239 Method A specifies the use of certified reference materials, or reference coals for calibration, the lab must employ a certified reference material or reference material traceable to a certified reference material for calibration verification that is not used for calibration.

A1.10.8.4 The lab selects calibration materials as well as a separate material for calibration verification from the list of available materials shown in Table A1.4.

A1.10.8.5 The lab cannot use either material A or material C as both of these materials have an expanded uncertainty greater than the reproducibility of the Test Method D4239.

A1.10.8.6 The lab must comply with the section on calibration in Test Methods D4239 Method A.

A1.10.8.7 The lab defines two calibration protocols.

A1.10.8.8 Protocol 1 is to use a fixed mass of calibrant materials A, B, D, E, G and H. The average calibration concentration is 2.19 % sulfur. The lab elects to employ material F for calibration verification.

TABLE A1.4 Calibrant Material Selection Criteria

Calibrant	Sulfur %	95 % Expanded Uncertainty	D4239 Reproducibility IR	Acceptable
A	0.380	0.055	0.054	NO
B	0.492	0.008	0.064	YES
C	1.010	0.120	0.111	NO
D	1.170	0.020	0.125	YES
E	1.475	0.029	0.153	YES
F	1.962	0.036	0.197	YES
G	3.076	0.031	0.297	YES
H	4.730	0.068	0.446	YES

A1.10.8.9 Protocol 2 is to use equally spaced masses of calibration material F to give the same instrument response as 200 mg of coal with concentrations from 0.50 to 4.00 % sulphur. The average calibration concentration is 2.25 % sulfur. The lab elects to employ material F for calibration verification.

A1.10.8.10 The instrument is calibrated employing both protocols to permit cross verification of results for test samples.

A1.10.8.11 The lab obtains 1.952 % for the verification material for protocol 1 and 1.985 % for the verification material for protocol 2. Since both verification runs agree with the assigned value within the reproducibility of the method for material F the lab can proceed with analysis of test samples.

A1.10.9 *Example 3—ASTM Test Method D3174:*

A1.10.9.1 ASTM Test Method D3174 does not include a section on calibration verification. Since D3174 does not include a section on calibration verification the lab must use a certified reference material or reference material traceable to a certified reference material for calibration verification. The lab identifies a reference material close to the expected concentration of the samples.

A1.10.9.2 Repeat verification according to the requirements above for each batch of test samples. Tests, such as ash analysis, analyze all samples in the batch at essentially the same time, rather than analyzing them in sequential order; therefore, anything that affects a verification sample will also effect the laboratory samples.

A1.10.9.3 If verification fails, reject all test results for the batch. Take corrective action to identify the assignable cause and recalibrate the instrument.

A1.11 Uncertainty of Measurement

A1.11.1 The uncertainty of measurement shall be determined for each test performed, unless the test precludes this determination (example Test Method D4749 for sieve analysis). While some standard methods include expectations for uncertainty, the laboratory shall verify that it is meeting the expectations of the standard method.

A1.11.2 Statistical analysis shall be used, such as ASTM Test Method E2554, and shall include the determination of the intermediate precision of test results.

A1.12 Control Charts

A1.12.1 Control charts shall be used to identify trend, bias, and other situations indicating the laboratory is in statistical control at the time of measurement.

A1.12.2 Quality control materials that should be control charted include, but are not limited to:

A1.12.2.1 Analysis Blank,
A1.12.2.2 Duplicate samples,
A1.12.2.3 Calibration verifications,
A1.12.2.4 Control samples/materials,
A1.12.2.5 Matrix Spike Recovery, and
A1.12.2.6 Relative Percent Duplicate Spike Difference.

A1.12.3 A minimum of one control charted item shall be employed for each unique test or process.

A1.12.4 A calibration blank check that does not agree with the mean calibration blank value ±3 times the calibration blank standard deviation renders the calibration suspect.

A1.12.5 Controls shall include warning conditions as well as out of control conditions.

A1.12.6 Warning conditions shall include but are not limited to:

A1.12.6.1 Warning limits at 95 % uncertainty (2 standard deviations) on control charts,
A1.12.6.2 Out of control conditions shall include but are not limited to:
A1.12.6.3 Control limits at 99 % uncertainty (3 standard deviations),
A1.12.6.4 Seven (7) consecutive values from a control material on one side of the central control chart line,
A1.12.6.5 Ten (10) out of eleven (11) values from a control material on one side of the central control chart line, and
A1.12.6.6 Seven (7) consecutive points that form a trend downward or upward in a control chart.
A1.12.6.7 A control point that does not violate any of the warning or out of control conditions constitutes an in control measurement system at a decision point, and
A1.12.6.8 A control point greater than or equal to the out of control limit constitutes an out of control measurement system at a decision point.

A1.12.7 In the case of a suspect measurement system verify whether the measurement system remains in a state of control.

A1.12.8 In the case of an out of control measurement system, flag all prior measurement results back to the last in control decision point as out of control. Take corrective action to bring the measurement system into a state of control. Analysis of test samples can proceed with the documented approval of a designated authority.

A1.13 Reporting Procedures

A1.13.1 The laboratory shall have procedures describing requirements for entry, verification, calculation, reporting, and approval of laboratory results.

NOTE A1.6—*Example of a Typical Practice*: The individual entering data or calculated results, or both shall not verify their own data entry or calculations. Only results from qualified personnel are to be included in a test report. In cases where the most current version of a method of a test is not employed provide the specific designation (including revision) of the method employed to generate the test result. In cases where a non-standard method of a test is employed provide the laboratory designation and last date of validation of the non-standard method of test employed to generate the test result. Include a statement that quality control data were generated and verified concurrent with test data and consistent with laboratory acceptance criteria. Include a statement that all quality control data and acceptance criteria are available for independent review and audit.

A1.14 Qualification of Laboratory Personnel

A1.14.1 The laboratory shall have procedures describing training, performance, verification and documentation criteria employed to qualify laboratory personnel to perform the sampling, sample preparation and test procedure capabilities claimed by the laboratory. This includes all associated maintenance, calibration and QA/QC.

A1.14.2 Document the identity of personnel as well as the most recent date of training or verification of qualification, or both.

NOTE A1.7—*Example of a Typical Practice*: Select benchmark performance criteria that are consistent with the intended use of the results reported by the laboratory. These can include but are not limited to the precision of standard test methods, regulatory batch quality control requirements, the Horwitz[7] function (recognized by IUPAC), and comparison with historical performance of qualified laboratory personnel. Employ pure substances, pure mixtures, certified reference material (CRMs), external reference materials (ERMs), internal reference materials (IRMs) and proficiency test samples (PTSs) to qualify personnel. Personnel are to test these materials as blinds.

A1.15 Environmental Conditions (Sampling, Sample Preparation and Analyses)

A1.15.1 Both temperature and humidity fluctuations effect sample preparation and testing process.

A1.15.2 Steps shall be taken to maintain a consistent humidity where this can be controlled by the laboratory.

A1.15.3 Steps shall be taken to maintain a consistent temperature where this can be controlled by the laboratory.

A1.15.4 Section 5.3 of ANSI/ISO/IEC 17025 provides more detailed requirements for maintaining environmental conditions.

[7] Horwitz Function—International Union of Pure and Applied Chemists (IUPAC, *Pure & Appl. Chem.*, Vol 62, No.1, 1990, pp. 149–162).