Designation: D7448 – 09

StandardPractice 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 ANS/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 ANS/ISO/IEC 17025 provided they do not invalidate the requirements of ANS/ISO/IEC 17025.

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

1.4 *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

D6349 Test Method for Determination of Major and Minor Elements in Coal, Coke, and Solid Residues from Combustion of Coal and Coke by Inductively Coupled Plasma—Atomic Emission Spectrometry

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E456 Terminology Relating to Quality and Statistics

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

2.2 *ANS/ISO/IEC Document:*

17025 General Requirements for the Competence of Calibration and Testing Laboratories[3]

3. Significance and Use

3.1 International standard ANS/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 ANS/ISO/IEC 17025 specifies the requirements for sound management. Clause 5 of ANS/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 ANS/ISO/IEC 17025, the Annex of this standard practice

[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 Quality Assurance.

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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.

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 ANS/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 ANS/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 ANS/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.
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.

ANS/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.

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$^{\varepsilon}$	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.

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, **(1)**[4] as well as ISO **(2, 3)** 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.

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." **(4)**

(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." **(4)**

(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:*

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.