Designation: D 4182 – 97

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

This standard is issued under the fixed designation D 4182; 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.

INTRODUCTION

Traditionally, coal testing facilities have been established to serve the specific interests or activities of one or more of the following:

(*1*) Exploration and evaluation of coal fields.

(*2*) Establishment of mining and coal preparation capabilities.

(*3*) Coal quality evaluation: (*a*) to meet contractual obligations; (*b*) to determine compliance with municipal, state, and federal regulations; and (*c*) to generate documentation used to establish power rates or rate basis.

(*4*) Confirmation of properties pertinent to coal processing and utilization, such as direct combustion or the production of coal-derived products (for example, metallurgical coke, carbons, liquids, and gas).

(*5*) Coal research pursuits.

1. Scope

1.1 This practice is limited to the evaluation of laboratories using ASTM procedures that are under the jurisdiction of Committee D-5 on Coal and Coke. They may be used to evaluate a laboratory's capability to perform the functions for which it has been established. It is not the intention that this practice be used to evaluate capabilities beyond those specifically claimed by the laboratory.

2. Referenced Documents

2.1 *ASTM Standards:*

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 409 Test Method for Grindability of Coal by the Hardgrove-Machine Method[2]

D 1989 Test Method for Gross Calorific Value of Coal and Coke by Microprocessor Controlled Isoperibol Calorimeters[2]

D 2013 Method of Preparing Coal Samples for Analysis[2]

D 2234 Practice for Collection of a Gross Sample of Coal[2]

D 2795 Test Methods for Analysis of Coal and Coke Ash[2]

D 2961 Test Method for Total Moisture in Coal Reduced to 2.36-mm (No. 8) Mesh Top Sieve Size (Limited-Purpose Method)[2]

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[2]

D 3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal[2]

D 3175 Test Method for Volatile Matter in the Analysis Sample of Coal and Coke[2]

D 3177 Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke[2]

D 3178 Test Methods for Carbon and Hydrogen in the Analysis Sample of Coal and Coke[2]

D 3179 Test Methods for Nitrogen in the Analysis Sample of Coal and Coke[2]

D 3286 Test Method for Gross Calorific Value of Coal and Coke by the Isoperibol Bomb Calorimeter[2]

D 3302 Test Method for Total Moisture in Coal[2]

D 3682 Test Method for Major and Minor Elements in Coal and Coke Ash by the Atomic Absorption Method[2]

D 4239 Test Methods for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods[2]

D 4326 Test Method for Major and Minor Elements in Coal and Coke Ash by X-Ray Fluorescence[2]

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[1] This practice is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.30 on Accreditation.

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[2] *Annual Book of ASTM Standards*, Vol 05.05.

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D 4621 Guide for Accountability and Quality Control in the Coal Analysis Laboratory[2]
D 5142 Test Methods for the Proximate, Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures[2]
D 5373 Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke[2]
D 5865 Test Method for Gross Calorific Value of Coal and Coke[2]
E 548 Guide for General Criteria Used for Evaluating Laboratory Competence[3]
E 1187 Terminology Relating to Laboratory Accreditation[3]
E 1267 Guide for ASTM Standard Specification Quality Statements[4]
E 1323 Guide for Evaluating Laboratory Measurement Practices and the Statistical Analysis of the Resulting Data[3]

3. Significance and Use

3.1 This practice provides guidelines to be followed by an individual or an accrediting body in evaluating a laboratory's capability to perform those functions only for which the laboratory claims competence. A laboratory for the purpose of this practice may be considered as any facility capable of carrying out one or more of the following functions relevant to coal or coke, or both:

3.1.1 Sampling,
3.1.2 Sample preparation,
3.1.3 Chemical analysis, and
3.1.4 Physical testing.

4. Functions

4.1 The laboratory personnel should be able to demonstrate a satisfactory knowledge of Practice D 2234 for coal or Practice D 346 for coke (or both), and the laboratory should have the capability to perform sampling in accordance with the appropriate practice.

4.2 The laboratory personnel should be able to demonstrate a satisfactory knowledge of Method D 2013 for coal or Practice D 346 for coke (or both), and the laboratory should have the capability to prepare samples in accordance with the appropriate method or practice.

4.3 The laboratory personnel should be able to demonstrate a satisfactory knowledge of all testing and analysis procedures for which the laboratory claims capability, and the laboratory should have the capability to perform those tests and analyses. The laboratory should be able to confirm that the specified equipment and reagents are available and that they are routinely used for those methods.

5. Guidelines for Selection of an Evaluator

5.1 The individual(s) selected to evaluate the competence of a laboratory to perform coal analyses by ASTM standards should be chosen primarily by experience. This evaluator should be familiar with the procedures required, as well as standard laboratory practices for quality assurance, and should have sufficient technical background to be able to comprehend the application of the appropriate procedures to the analysis of coal, coke, or coal ash. The competent evaluator should be able to communicate effectively, both orally and in writing, and should be familiar with the criteria against which the laboratory is to be evaluated.

6. Deviation from Standard Method

6.1 Documentation must be available to demonstrate that any procedures or practices that deviate from those specified in ASTM standard procedures must yield results equivalent to the appropriate ASTM standard procedures.

7. Checklist

7.1 To provide uniformity, the evaluator should review the laboratory with the aid of a worksheet or guideline. An example is provided in Appendix X2. Any worksheet or checklist used by the examiner should include at least those areas covered by Sections 1 through 7 of Appendix X2. Examples are not provided for all test methods that are under the jurisdiction of ASTM Committee D-5, nor is it the intention of ASTM Committee D-5 to provide checklists for all tests and analyses that are under its jurisdiction. The examples provided may be used as templates for checklists that can be used to evaluate tests not covered by the example checklist.

7.2 Comments made by the evaluator should be keyed to item numbers in the checklist and may be made on a separate sheet, on the margins, or on the reverse of the checklist.

7.3 The numbering of the notes to accompany the example checklist that are presented in Appendix X1 corresponds to that of the checklist. The notes describe most of the procedures that define a capable laboratory and may be used by a laboratory as a guide to establishing quality procedures and good laboratory practices.

8. Keywords

8.1 audits; calibration; certification; evaluation; laboratory; quality assurance; quality control; quality manual; quality system; traceability

[3] Annual Book of ASTM Standards, Vol. 14.02.
[4] Withdrawn. See 1995 *Annual Book of ASTM Standards*, Vol 14.02.

APPENDIXES

(Nonmandatory Information)

X1. NOTES TO ACCOMPANY THE EXAMPLE CHECKLIST (Appendix X2)

X1.1 Quality Assurance Program

X1.1.1 There should be a Quality Assurance (QA) Manual available that documents all of the components of the quality system (see Guide E 548, Sections 3.1.11 and 6.2).

X1.1.2 The QA Manual should be updated whenever any quality system provisions are changed. A section of the manual should make explicit the procedures to be followed for making the modifications and should specify who has the authority to make them.

X1.1.3 A master document control list that defines the current revision status of all referenced documents, practices, procedures, and standards used in the laboratory should be maintained and made readily available throughout the laboratory.

X1.1.4 The QA Manual should contain a summary of the QA program and

X1.1.5 A written statement of management commitment to the QA program.

X1.1.6 A QA Coordinator or Manager (sometimes assisted by a QA Committee) should be responsible for assuring that the QA program is in place and working. Generally, the Coordinator should report directly to the manager of the laboratory.

X1.1.7 The laboratory should maintain an up-to-date list of all of the sampling, preparation, testing, and analysis procedures for which it claims competence.

X1.1.8 The QA Manual should include or reference a written Standard Operating Procedure (SOP) for each procedure and test that is conducted by the laboratory (Note X1.1). Though ASTM standards should be referenced, if the laboratory deals with international customers, international standards may be referenced. If a procedure deviates from an established standard, data must be available to demonstrate that the modified procedure provides results that are equivalent to those obtained by using the standard. Simple reference to published standards, however, is generally inadequate; such reference needs to be supplemented by additional information pertaining to quality assurance and quality control activities that are frequently not covered in test procedure standards.

NOTE X1.1—The terms "Standard Operating Procedure" and "SOP" are used in this document to designate any written procedure whether it applies to a specific test procedure or a procedure that is used in another aspect of the laboratory operation including quality elements. The laboratory may refer to such documents by a name other than Standard Operating Procedure.

X1.1.9 Calibration is the set of operations that establish, under specified conditions, the relationship between values indicated by a measuring instrument, measuring system, or values represented by a material measure and the corresponding known values of a measurand (Guide E 548).

X1.1.9.1 Where appropriate, procedures employed for calibrating the equipment used should be defined in an SOP. It should include a value for an acceptable deviation and procedures for dealing with results that exceed the acceptable deviations.

X1.1.9.2 A schedule should be maintained that designates when calibration is to be performed on each piece of equipment that requires it (see X1.1.11 following).

X1.1.9.3 Procedures should be defined for establishing traceability of reference materials (RMs, Note X1.2), including in-house control samples (Guide D 4621, Section 3.1.7 and Appendix X2), to certified reference materials (Note X1.3). See also Guide E 548, Section 3.1.13 and Guide E 1267, Sections 3.2.2 and 5.2.2 for discussions of traceability.

NOTE X1.2—Reference material: a material or substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to a material (Terminology E 1187).

NOTE X1.3—Certified reference material: a reference material for which one or more property values are certified by a technically valid procedure accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.

X1.1.10 Procedures for dealing with instances of nonconformance when analyzing RMs or control samples should include the following issues:

X1.1.10.1 The SOP should define statistically based criteria for recognizing nonconformance, including numerical limits that require action when exceeded (namely, "action limits," Guide D 4621, Appendix X1). See also Guide E 1323 for guidance on statistical analysis practices.

X1.1.10.2 It should also define specific procedures to be followed to investigate the cause of nonconformance.

X1.1.10.3 It should define procedures to correct the SOP or equipment.

X1.1.10.4 A description of nonconformance occurrences and actions taken should be documented in a permanent manner.

X1.1.10.5 Procedures should be defined to validate that any modification does accomplish the objective and that the process is under control.

X1.1.10.6 Specific procedures should be modified or instituted to prevent recurrence of the nonconformance.

X1.1.10.7 Authority to make modifications of equipment or SOPs should rest with only one designated person (or, at most, only a few persons). This policy should be enforced to prevent casual tampering with or modification of equipment and procedures, especially when nonconforming results are detected. Authority to appoint deputies or substitutes should reside only with one individual.

X1.1.11 There should be a calendar made up in advance that lists the routine QA activities that will be undertaken, such as recalibration, audits, equipment maintenance, and so forth.

X1.1.12 There should be a written policy for continuous improvement.