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Standard Practice for Production of Coal, Coke and Coal Combustion Samples for Interlaboratory Studies¹

This standard is issued under the fixed designation D6796; 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.

^{ε1} NOTE—Editorial corrections were made to X1.1.1.13, X1.1.1.14, and X2.2.2 in June 2021.

1. Scope

1.1 This practice describes the essential activities that are required to produce samples for interlaboratory studies.

1.2 The suitability of a particular interlaboratory sample developed using this guide will depend on the tests being made.

1.3 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D121 Terminology of Coal and Coke

D346/D346M Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

D2013/D2013M Practice for Preparing Coal Samples for Analysis

D2234/D2234M Practice for Collection of a Gross Sample of Coal

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.

D2492 Test Method for Forms of Sulfur in Coal (Withdrawn 2021)³

D2961 Practice for Single-Stage Total Moisture Less than 15 % in Coal Reduced to 2.36 mm [No. 8 Sieve] Topsize

D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke

D3302/D3302M Test Method for Total Moisture in Coal

D4596 Practice for Collection of Channel Samples of Coal in a Mine

D5192 Practice for Collection of Coal Samples from Core

D5263 Test Method for Determining the Relative Degree of Oxidation in Bituminous Coal by Alkali Extraction

D7430/D7430M Practice for Mechanical Sampling of Coal

D7582 Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis

E178 Practice for Dealing With Outlying Observations

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E826 Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D121.

4. Summary of Practice

4.1 Test samples of adequately defined composition and homogeneity are required for interlaboratory studies. These samples should be typical in all respects to the samples for which the test method is applicable.

4.1.1 The sample should be of sufficient quantity to perform all necessary tests and replicates. Guidelines specifying minimum quantities are given in Practice D2013/D2013M.

4.1.2 The top size must be consistent for the test being run.

4.2 Differences in results due to the homogeneity or oxidation, or both, of the test sample should be minimal with respect to other errors.

³ The last approved version of this historical standard is referenced on www.astm.org.

5. Significance and Use

5.1 The objective of this practice is to provide guidelines for the preparation of samples for interlaboratory studies to evaluate new test methods or for quality control purposes.

5.2 Samples prepared using these guidelines may be used for the testing of the precision and bias mandatory for ASTM test methods. Such an evaluation is necessary to provide guidance to the user as to the reliability of measurements that can be expected by its use. The statements are developed on the basis of user experience (ordinarily through interlaboratory studies) with the test method.

5.3 The availability of appropriate samples is a key requirement for interlaboratory studies.

5.4 The homogeneity of the sample produced for interlaboratory studies must be small enough so that the variance caused by the sample is small compared to the variance of the test being performed.

6. Sample Selection and Preparation

6.1 Collect samples using Practices [D2234/D2234M](#), [D4596](#), [D5192](#), and [D7430/D7430M](#) for coal, or Practice [D346/D346M](#) for coke. Coal combustion products should be sampled in a manner to provide a representative sample free from contamination. The sample should be typical of a certain source and class of samples (for example, Eastern U.S. bituminous coal) or a certain value for a component (such as coals with two percent sulfur), or both, so that appropriate diversity of samples can be identified for interlaboratory studies. See [Note 1](#). To ensure diversity, verify the important values and the rank of coal samples through additional testing prior to distribution.

6.2 Grind samples to the proper size required by the test method and properly homogenize and split. Prepare samples using Practice [D2013/D2013M](#). Some coal combustion products may not require grinding, but tests should be run to determine if further grinding is necessary.

6.3 Both single-laboratory precision and between operator-laboratory precision should be evaluated. Material of adequate homogeneity but of uncertain composition may be used to determine precision of a given test method.

NOTE 1—The use of materials of demonstrated adequate homogeneity and known value for the test enables evaluation of the precision and bias of a test method. Materials of known value for the test are required to identify biases. Consensus values obtained as the result of interlaboratory studies are not adequate to permit quantitative evaluation of biases, although they may show that such exist.

6.4 Use six or more materials for each interlaboratory study. See [Notes 1-3](#).

6.5 Each laboratory shall receive its own sample and at least one sample shall be archived by the interlaboratory study coordinator.

6.6 The bulk sample should be available in sufficient quantity for the requirements of the interlaboratory study, to test for homogeneity and to permit further examination by other techniques.

6.7 Sample preparation and distribution should provide enough material and time so that laboratories can perform test runs on new or unfamiliar methods. This is very important because only laboratories that are thoroughly familiar and experienced with the procedure can be considered qualified to participate in the interlaboratory study.

6.8 Sample stability (see [7.6](#)), storage, and future accessibility should be considered.

NOTE 2—Practice [E691](#) paragraph 10.2.2 states, “An ILS of a test method should include at least three materials representing different test levels, and for development of broadly applicable precision statements, six or more materials should be included in the study.” Because of the peculiarities of different ranks of coal, more than the minimum six materials are needed to develop precision statements applicable to all coal types. Under no circumstances should the final statement of precision of a test method be based on acceptable test results for each material from fewer than 6 laboratories.

NOTE 3—In preparing an interlaboratory sample, the range of values of the property being measured should be considered, the ranks of coal to which the method will be applied, the difficulty and expense of preparing and distributing samples, the length of time and expense in performing the test and the commercial or legal need for reliable estimates of precision when choosing the number of materials to be used.

7. Determining the Suitability of the Sample for the Test Method

7.1 After the sample has been prepared, or if a previously prepared sample is to be used for tests other than for what it was originally prepared, the homogeneity of the sample must be determined.

7.2 Test samples must be sufficiently homogeneous to evaluate the test method. The degree of homogeneity is related to the size of the sample analyzed. Hence the minimum size of a sample meeting a specified homogeneity must be stated, and this must be equal to or smaller than the size of the sample specified in the test method. Also, different tests may require different sample sizes to achieve homogeneity. Tests for homogeneity should be made. An acceptable method for measuring homogeneity is given in [Appendix X1](#).

7.3 The uncertainties in homogeneity, moisture, or sample composition, or a combination thereof, should not exceed one third of the measurement uncertainties they are expected to evaluate. When this specification is met, sample uncertainties make a negligible contribution to the variance of the test results. While replicate samples may be used in practical analysis to average out sample variations, this is not feasible in the case of samples for Interlaboratory Studies; hence, homogeneity should be attained as far as possible.

7.4 Samples not meeting the homogeneity requirements above for a given test should not be used in interlaboratory studies unless they are re-mixed (all samples must be recombined and the entire sample rehomogenized, see [6.2](#)) and retested. In some cases, samples that are inhomogeneous for one test may be homogeneous for other tests.

7.5 Top size of the sample must be consistent with required top size or reduced to the appropriate size.

7.6 Sample stability may be determined. See [Note 4](#).

7.6.1 *Moisture*—For most tests, moisture should be determined and compared to other results and calculated to a common basis.

7.6.2 *Oxidation*—Coals (particularly those of low rank) stored for a long period of time may become oxidized. If pyrite becomes oxidized, it may affect forms of sulfur determinations (see Test Method [D2492](#)). Bituminous coals are generally stable for long periods of time, but the degree of oxidation of bituminous coals can be determined using Test Method [D5263](#).

8. Sample Storage and Preservation

8.1 Samples should be stored in such a way to maintain sample stability for the characteristics being measured. See [Note 4](#).

8.1.1 Changes of values in ultimate analysis should be less than 1 % over a 10 year period.

8.1.2 Moisture should be determined by an appropriate method (Test Method [D3173](#), Test Methods [D7582](#), Test Method [D3302/D3302M](#), or Practice [D2961](#)) prior to each analysis. It should not be assumed that moisture content will remain constant from day to day especially for low rank coals.

8.1.3 Except for moisture, the largest absolute differences in parameter value changes are likely to be for percent carbon due to oxidation.

8.1.4 For less stable coals or coals with high moisture contents, or both, more aggressive storage or preservation procedures may be employed. See [Note 5](#).

8.2 Samples may be conditioned (preoxidized) prior to splitting and bottling. See [Appendix X2](#).

8.2.1 Samples may be stored under nitrogen or argon to minimize oxidation. Nitrogen is cheaper but argon is less likely to react with coal components.

8.2.2 Samples may be refrigerated to minimize oxidation. Samples should be maintained at about 4 °C. Care must be taken to minimize changes in moisture. It is not recommended that samples be frozen as this might change to pore structure of the coal.

8.2.3 Depending on the characteristics of the coal to be measured, high moisture coals may be stored in water.

NOTE 4—Storing samples in darkness or brown bottles can reduce photochemical changes. Air-dried relatively stable-low moisture coals, coke, and stable combustion products can be stored in tightly sealed glass or plastic containers at room temperature for long periods of time without significant change of most coal characteristics.

NOTE 5—Low rank coals (subbituminous and lignite) generally have high moisture content and oxidize rapidly. This oxidation may continue for months but will slow with time. Moisture values in these samples may depend on the time the sample is taken and the relative humidity of the laboratory and may change within seconds after the sample is removed from the bottle and over a longer period of time within the bottle.

9. Record Keeping

9.1 Characteristics of the sample should be recorded. At a minimum, the rank, percent ash, top size, approximate moisture content, special handling comments, amount of sample available, and original purpose for which the sample was collected shall be recorded, that is, sample location, sample type (run of mine; Terminology [D121](#), channel sample; Practice [D4596](#), etc). Maintain all results with the samples until they are published, at which time only the reference need be maintained.

9.2 Record when the sample was collected, record when the sample was prepared, and record how the sample has been maintained.

9.3 A list of samples currently available with the information listed in [8.1](#) and [8.2](#) should be accessible to interested parties who may wish to use them for interlaboratory studies.

10. Keywords

10.1 ash; coal; coke; combustion residue; homogeneity; laboratory; moisture; oxidation; quality control

APPENDIXES

(Nonmandatory Information)

X1. TEST FOR HOMOGENEITY

X1.1 Test for homogeneity shall be conducted by a single laboratory in a manner similar to that given in Practice [E826](#) prior to sample distribution.

X1.1.1 Conduct all tests by the best available procedure under optimum conditions. Preferably a method with a known precision and bias shall be used.

X1.1.2 If the samples have been split into less than 15 laboratory samples, sub-samples from each laboratory sample shall be measured for the test of interest.

X1.1.2.1 Whenever possible, the test portions should be taken only after the laboratory samples is rolled in its container at least 5 turns followed by inverting the sample 5 times.

(1) This process should be repeated at least 3 times.

(2) A sample can then be taken from the top.

X1.1.2.2 The steps in [X1.1.2.1](#) should be repeated for all subsequent test portions even if they are taken from the same container.

X1.1.3 If the number of containers is so large that test portions from each container cannot be measured, a minimum of 8 %, but not less than 15 %, should be tested for homogeneity.

X1.1.4 Label all containers consecutively (that is, 01, 02, 03, ...c). If all sample containers will not be measured (see [X1.1.3](#)), select the containers to be measured as follows: From a table of random numbers (see [Table X1.1](#) and the Warning note below) pick an arbitrary starting place and select any

