



Standard Practice for Preparing Coal Samples for Analysis¹

This standard is issued under the fixed designation D2013/D2013M; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This practice² covers the reduction and division of gross or divided samples, and the preparation of composite samples, up to and including the individual portions for laboratory analysis.

1.2 Reduction and division procedures are prescribed for coals of the following groups:

1.2.1 *Group A* includes coals that have been cleaned in all sizes.

1.2.2 *Group B* includes all other coals. Unknown coals are to be considered under Group B.

1.2.3 Group A allows smaller weight laboratory samples to be retained than Group B. These lower weights may be used for particular coals if they have been shown, by using the procedures of **A1.2**, to give a sample preparation and analysis variance which is no more than 20 % of the total analysis variance.

1.3 *Units*—The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

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.*

¹ This practice is under the jurisdiction of ASTM Committee **D05** on Coal and Coke and is the direct responsibility of Subcommittee **D05.23** on Sampling.

Current edition approved Oct. 1, 2021. Published December 2021. Originally approved in 1962. Last previous edition approved in 2020 as D2013/D2013M – 20. DOI: 10.1520/D2013_D2013M-21.

² For more detailed explanation of this practice, see Keller, G. E., “Determination of Quantities Needed in Coal Sample Preparation and Analysis,” *Transactions*, Vol 232, 1965, pp. 218–226.

2. Referenced Documents

2.1 *ASTM Standards*:³

D121 Terminology of Coal and Coke

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

D3173/D3173M Test Method for Moisture in the Analysis Sample of Coal and Coke

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

D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases

D3302/D3302M Test Method for Total Moisture in Coal

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

D7430 Practice for Mechanical Sampling of Coal

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

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

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

E456 Terminology Relating to Quality and Statistics

2.2 *ISO Standard*:⁴

ISO 13909 Hard Coal and Coke—Mechanical Sampling

3. Terminology

3.1 *Definitions of Terms Specific to This Standard*—No terms are used which are specific to this practice. Many terms used in this practice may be found in Terminologies **D121** and **E456** and in Practice **E177**.

4. Summary of Practice

4.1 Three processes of sample division and reduction are covered as follows:

³ 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.

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>.

4.1.1 *Procedure A*—Manual riffles are used for division of the sample and mechanical crushing equipment for the reduction of the sample.

4.1.2 *Procedure B*—Mechanical sample dividers are used for the division of the sample and mechanical crushing equipment for the reduction of the sample.

4.1.3 *Combined Procedure A and B*—The two procedures may be combined at any stage.

5. Significance and Use

5.1 Other standards are used to collect the gross sample: Practice **D2234/D2234M** allows for one division of the gross sample before crushing. The mass and top size of the gross or divided sample collected by using these guides and practices are usually too large for chemical or physical testing. Practice **D2013/D2013M** provides instructions for reducing and dividing the gross or divided sample, by on-line or off-line processes, or both, to a top size and mass suitable to the performance of testing. Any bias in the gross or divided sample before adherence to this practice will remain in the final sample resulting from use of this practice. Therefore, carefully select the standard to be used to collect the gross sample.

5.2 Division and reduction of a sample may occur at more than one location. Most often, the sample is collected, reduced, and divided (one or more times) by use of a mechanical sampling system. The remaining sample may be further divided on-site to facilitate transporting it to the laboratory where further reduction and division likely occurs before analysis.

5.3 In places, this practice requires air drying the sample before subsequent reduction. Procedures for air drying and air-dry loss determination are provided in Test Method **D3302/D3302M**.

5.4 Most often, samples are reduced and divided to an analysis sample. However, some tests may require a sample of different mass or top size. This procedure may be used to provide a sample of any mass and size consist from the gross or divided sample to, and including, the analysis sample.

5.5 This practice also specifies how to prepare composite samples, if required.

6. Apparatus

6.1 *Crushers or Grinders*—Jaw, cone, or rotary crusher; hammer mill; roll; or other suitable crusher to reduce the sample to pass the sieve designated in 6.4. Hard steel or chilled iron plate with tamper, sledge, or hand bar may be used for preliminary crushing of any large lumps in the sample before feeding into the crusher. Crushers should be designed and operated in a manner to minimize the effect of induced air circulation and thus the potential for drying the coal.

6.1.1 *Pulverizer or Mill*—For final reduction of laboratory sample to the 250 μm [No. 60] sieve size, the following equipment may be used:

6.1.2 *Hammer Mill*—Completely enclosed to avoid loss of dust or moisture.

6.1.3 *Porcelain-Jar Ball Mill*—This mill shall be approximately 230 mm [9.0 in.] in diameter and 250 mm [10.0 in.] in

height with smooth, hard, well-rounded, flint pebbles, or equivalent, that do not increase ash content of the sample.

6.1.4 *Bucking Board (Chrome Steel) or Mortar (Agate or Equivalent) and Pestle*—Only for reducing the small fraction of sample, not passing a 250 μm [No. 60] sieve after pulverization.

6.2 Sample Dividers:

6.2.1 *Mechanical*—A mechanical sample divider using a reciprocating or rotating cutter, a rotating hopper and spout, a rotating slotted cone, a reciprocating hopper and fixed cutter, bucket cutter with either bottom dump or inverting discharge, slotted belt, rotary disk divider, mechanical stopped or moving belt sweeper, or other acceptable devices for dividing the sample. Typical mechanical sample dividers are shown in **Fig. 1**. These illustrate various designs, but other acceptable designs are available.

6.2.2 *Riffle*—A manual sample divider that splits the coal stream into two parts when it is directed through alternating slots. Riffle slots shall be at least three times the top size of the coal being divided and have an even number of alternating slots, in order to achieve similar mass delivered to each catch pan. Riffles shall be enclosed. The slope of the feed chutes and riffles shall be at least 60°.

6.2.2.1 *Large Riffle*—A manual sample divider with at least fourteen 12.7 mm [$\frac{1}{2}$ in.] to 25.4 mm [1 in.] divisions for dividing subsamples of coal passing 2.36 mm [No. 8], and 4.75 mm [No. 4] sieves. See **Fig. 2**.

6.2.2.2 *Small Riffle*—A manual sample divider with at least twenty-four 6.35 mm [$\frac{1}{4}$ in.] to 9.52 mm [$\frac{3}{8}$ in.] divisions for dividing the subsamples of coal passing 250 μm [No. 60]. See **Fig. 3**.

6.2.2.3 *Feed Scoop*—A feed scoop or pan having straight sides and a width equal to the effective width of the riffle shall be used to feed the riffle.

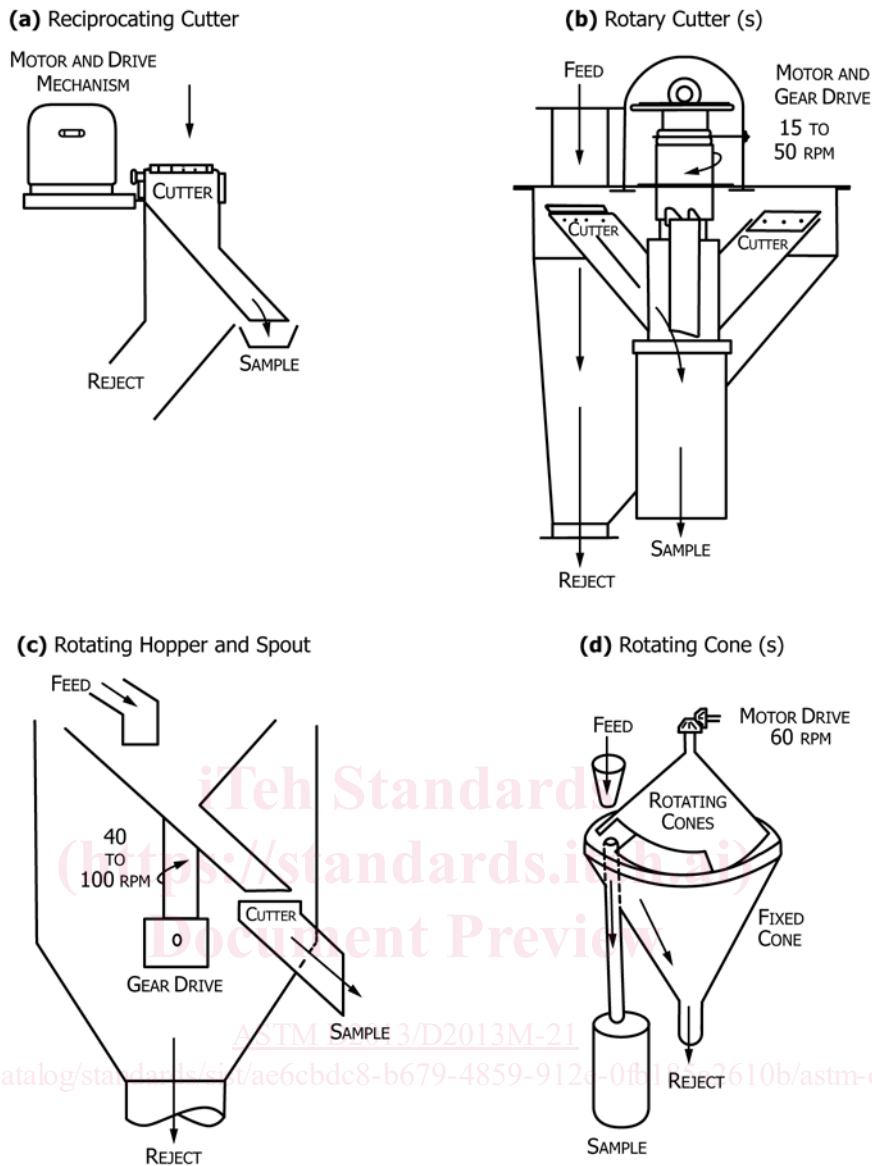
6.2.2.4 *Feed Chute*—A feed chute shall be used as shown in **Fig. 2**. The minimum discharge opening of the feed chute shall be the same width as the riffle slot opening.

6.3 *Mixing Wheel*—One type of a mechanical device used for mixing the analysis sample. In this device, the samples are in closed containers attached to the rim of a wheel at an angle of 45° with the horizontal wheel shaft. The wheel provides space for a number of containers depending on its diameter and is turned slowly by a small motor and reduction gear. The wheel should be rotated at a speed so that the particles fall gently from top to bottom of the container, mixing the sample thoroughly. The container should be about half full and never more than two-thirds full to obtain good mixing of the sample.

6.4 *Sieves*—A set of sieves whose dimensions are in accordance with Specification **E11**, of the following sizes, with cover and receiver:

| No. | Size |
|-----|-------------------|
| 4 | 4.75 mm |
| 8 | 2.36 mm |
| 20 | 850 μm |
| 60 | 250 μm |

6.5 *Laboratory Sample Containers*—Heavy vapor-impermeable bags, properly sealed, or noncorroding cans such as



(a) *Reciprocating Cutter*—Fig. 1(a) shows a section of a cutter which is moved across a stream of coal. At regular intervals, the cutter movement is reversed and a sample increment is collected on each trip through the coal stream.

(b) *Rotating Cutter*—Fig. 1(b) shows two cutters attached to a hollow, rotating shaft. Each cutter is designed to extract increments from the feed and to discharge these into the hollow shaft. One or more cutters may be used.

(c) *Rotating Hopper and Spout*—Fig. 1(c) shows the totaling hopper that receives the crushed sample and discharges it through a spout over one or more stationary cutters.

(d) *Rotating Cone*—A sampler developed by the British National Coal Board. Two slotted cones are locked together and rotated on a vertical shaft so that on each revolution the common slot operating intercepts the falling stream of coal and collects an increment.

FIG. 1 Mechanical Sample Dividers

those with an airtight top, friction top, or screwtop sealed with a rubber gasket and pressure-sensitive tape for use in storage and transport of the laboratory sample. Glass containers, sealed with rubber gaskets, may be used, but care must be taken to avoid breakage in transport.

7. Precautions

7.1 *General*—The preparation of the gross or divided sample, or the composite sample, shall be performed by, or under the direct supervision of, personnel knowledgeable of proper sample handling practices. Sample preparation should

be checked at intervals by the methods described in [Annex A1](#) or [Annex A2](#). It is necessary that the variance of sample division and analysis S_{da}^2 be not more than 20% of the total variance of sampling, division, and analysis S_o^2 .

7.1.1 The sample preparation operations should be performed in an enclosed space, roofed, cool, and free from excessive air movements.

7.2 *Number of Tests*—Before preparing the gross or divided sample, or the composite sample, consider the number and nature of the analysis and tests to be performed. A separate

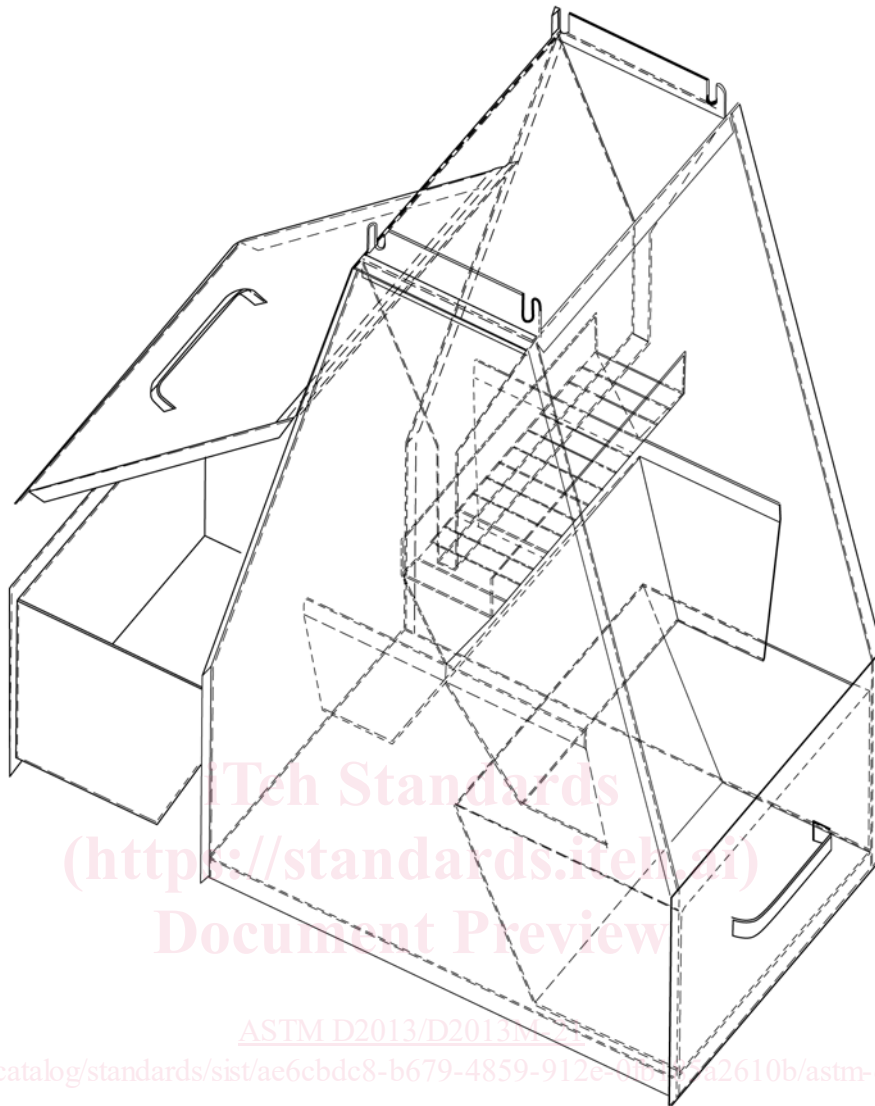


FIG. 2 Large Sample Divider (Riffle)

moisture laboratory sample may be required, and portions may be required for grindability and other tests. Also, a reserve sample may be desired in case a check analysis or test is required.

7.3 This practice specifies situations when air drying the sample is necessary during sample preparation. Test Method [D3302/D3302M](#) specifies procedures for air drying and calculation of percent air dry loss.

7.3.1 Calculate and record air-dry loss determination each time air drying is performed.

7.3.2 In handling, reducing, and dividing the sample, all operations shall be done rapidly and in as few operations as possible, since moisture loss depends on several factors other than total moisture content, such as time required for crushing, atmospheric temperature and humidity, and type of crushing equipment.

7.3.3 While awaiting preparation, the gross or divided sample shall be protected from moisture change as a result of exposure to rain, snow, wind, and sun on contact with absorbent materials.

7.3.4 Whenever subsamples are stored or transported, the containers and subsample shall be weighed and equilibrated to the new atmosphere by air drying, and the weight loss or gain shall be used in the calculation of moisture content.

7.4 Whenever a distinct change of humidity occurs during the course of preparation of an air-dried subsample, the subsample shall be weighed and its moisture equilibrated with the new atmosphere, and the loss or gain in mass shall be used in the calculation of moisture content.

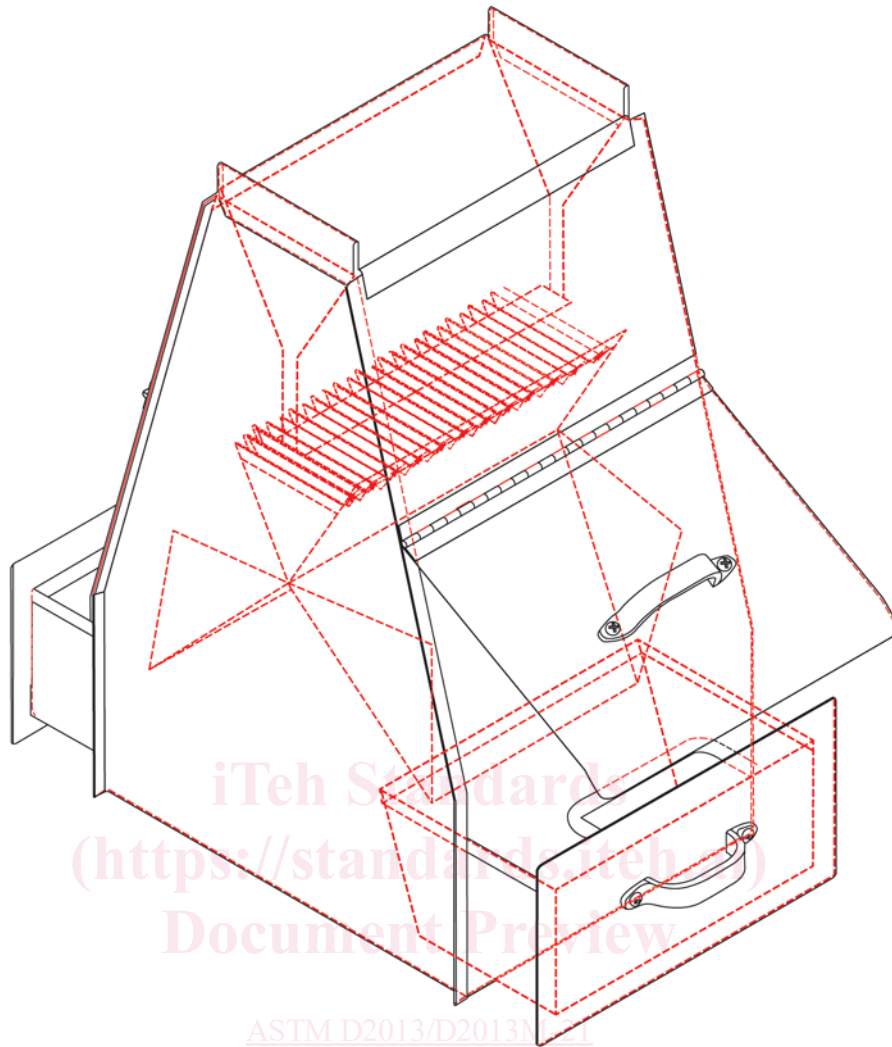


FIG. 3 Small Sample Divider (Riffle)

8. Sieve Tests

8.1 The errors of sample division are sensitive to the top size, and therefore, it is important to make a periodic sieve test of the product of the sample crusher. Sieve tests shall be made and reported in accordance with Test Method D4749.

9. Procedure

9.1 *Mass*—The minimum allowable mass of the sample at any stage depends on the top size, the variability of the constituent sought, and the degree of precision desired (Table 1).

9.2 *Reduction and Division* (See Fig. 4 for flowchart):

9.2.1 It is permissible to air dry the sample before crushing. Samples may require air drying to feed properly through the reduction and dividing equipment. Sometimes there is an interest in determining the air-dry loss value before crushing. Air dry in accordance with Test Method D3302/D3302M.

9.2.2 In the reduction and division of gross or divided samples for which total moisture content is to be determined, the precautions in 7.3 and 7.4 must be followed.

9.2.3 *Procedure A—Manual Riffing:*

TABLE 1 Preparation of Laboratory Sample

| Crush to pass at least 95 % through sieve | Divide to a minimum weight of, g ^A | |
|---|---|---------|
| | Group A | Group B |
| 4.75 mm [No. 4] | 2000 | 4000 |
| 2.36 mm [No. 8] | 500 | 1000 |
| 850 μm [No. 20] | 250 | 500 |
| 250 μm [No. 60] (100 % through) | 50 | 50 |

^A If a moisture sample is required, increase the quantity of No. 4 [4.75 mm] or No. 8 [2.36 mm] sieve subsample by 500 g.

9.2.3.1 Reduce the gross or divided sample to a top size of 4.75 mm [No. 4] or 2.36 mm [No. 8] sieve, taking precautions in accordance with Section 7.

9.2.3.2 Determine the number of passes required in the riffing operation from the total mass of the gross sample and the minimum permissible mass in accordance with Table 1.

9.2.3.3 Divide the crushed sample by using a large riffle. Riffles properly used will reduce sample variability but cannot eliminate it. A typical enclosed riffle is shown in Fig. 2 and described in 6.2.2. Pass the coal through the riffle from a feed

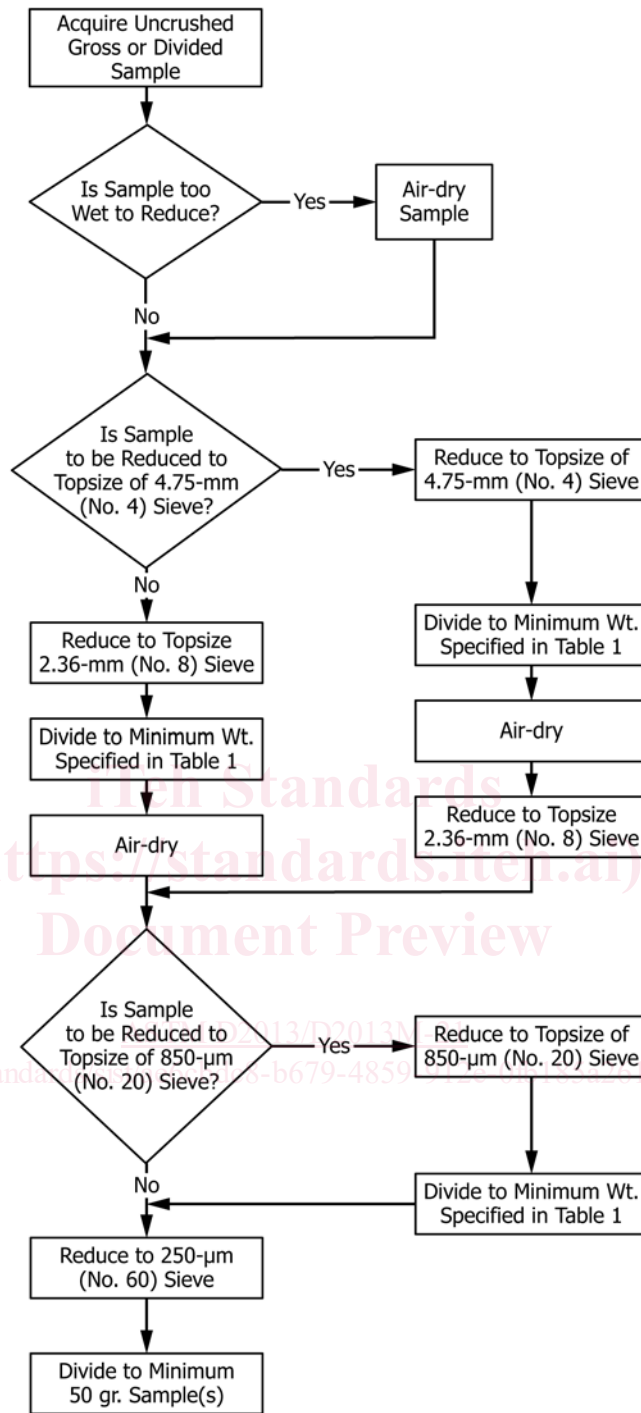


FIG. 4 Sample Preparation Flowchart

scoop, feed bucket, or riffle pan having a lip or opening the full width of the riffle. When using any of the preceding containers to feed the riffle, spread the coal evenly in the container, raise the container, and hold it with its front edge resting on top of the feed chute, then slowly tilt it so that the coal flows in a uniform stream through the hopper straight down over the center of the riffle into all the slots, then into the riffle pans, one half of the sample being collected in each pan. Under no circumstances shovel the sample into the riffle or dribble into the riffle from a small-mouthed container. Do not allow the

coal to build up in or above the riffle slots. If it does not flow freely through the slots, shake or vibrate the riffle to facilitate even flow.

9.2.3.4 If the initial crushing was only to 4.75 mm [No. 4] sieve size, reduce to 2.36 mm [No. 8] sieve size after dividing to no less than the quantity specified in Table 1 for a 4.75 mm [No. 4] sieve size.

9.2.3.5 After reducing to 2.36 mm [No. 8] sieve size, divide the subsample by riffing to no less than the quantity specified in Table 1 for a 2.36 mm sieve size.

9.2.3.6 With suitable pulverizing equipment (see 6.1), reduce the 2.36 mm [No. 8] sieve size subsample to a 250 μm [No. 60] sieve size. Divide the ground subsample by riffing, using the small riffle (see 6.2.2) until a minimum of 50 g is obtained. Quickly pass the subsample through a 250 μm [No. 60] sieve. Reduce the particles retained on the screen, on a bucking board or mortar and pestle to pass the sieve, and add to what passed through the sieve and mix thoroughly. This is the analysis sample.

9.2.3.7 As an alternative to the procedure of 9.2.3.6, the 2.36 mm [No. 8] sieve size subsample may be reduced to pass 95 % through a 850 μm [No. 20] sieve. Divide this subsample by riffing with the small riffle to not less than the quantity specified in Table 1, and then reduce to 250 μm [No. 60] sieve size in accordance with 9.2.3.6.

9.2.3.8 Thoroughly mix, preferably by mechanical means, the analysis sample, weighing not less than 50 g, before extracting portions for analysis (see 6.3).

9.2.4 Procedure B—Mechanical Division:

9.2.4.1 Reduce the gross or divided sample in stages and divide by suitable mechanical sample dividers (see 6.2.1) to quantities not less than those shown in Table 1.

9.2.4.2 Mechanical division of the sample consists of automatically collecting a large number of increments of the properly reduced sample. Distribute this large number of increments equally throughout the entire discharge from the sample crusher because crushers can introduce appreciable segregation. At each stage of division, take at least 60 increments.

NOTE 1—It is recommended that, in the case of mechanical division in which an increment is not thoroughly mixed with other increments before division, a portion of each increment be collected by the subsequent stage increment collection process.

9.2.4.3 Thoroughly mix the analysis sample, 100 % through 250 μm [No. 60] sieve and weighing not less than 50 g, in accordance with 9.2.3.8 before extracting portions for analysis.

10. Preparation of Composite Samples to Represent Lot-Size (or Consignment-Size) Quantities of Coal

10.1 There are several issues to consider when deciding whether to make a physical composite of separate samples collected to represent different parts of the same lot (or consignment). See Note 2. When the analytical parameters of interest are additive (for example, proximate and ultimate analyses), it is not unusual to test the individual samples and perform a ton-weighted mathematical average to determine the result for the lot (or consignment). See Note 3. On the other hand, when the sought-after parameters are non-additive (for example, ash fusibility, Hardgrove grindability, and rheological properties), analysis of a composite sample is the only way to achieve a meaningful test result. Another consideration is that since there are no provisions in this standard for dividing samples of top size larger than 250 μm [No. 60] by means other than riffing or mechanically subsampling, the exact masses needed for preparation of composite samples containing larger particles are not attainable using those processes alone. It is also important to realize that making a composite can sometimes result in unintended moisture changes after which the

percent moisture and other as-received basis data determined on the composite may differ slightly from the weighted average of the individual samples. In addition, errors associated with extraction of increments from multiple samples are unknown and can vary from sample to sample and with the absolute level of each analyte. Taking these and other issues into account leads to the requirement that preparation of composites be performed in accordance with the procedures described below. The details pertaining to the preparation of composite samples should be agreed to by all concerned parties.

NOTE 2—As used in this standard, the term *represent* (together with its related forms) does not imply that samples satisfy a particular quantitative or statistical relationship to the coal that was sampled. The term is used within Practice D2013/D2013M in the sense of its broad dictionary definition, that is, to simply stand for or symbolize larger quantities of coal.

NOTE 3—As used in this standard, parameters that are additive are those having values not affected by interactions between the physical and chemical properties of the combined individual samples. Non-additive parameters are those for which such interactions may occur or those for which definitive information on interactions is not available.

10.2 If two or more samples have been collected by a single mechanical coal sampling system operated under constant settings, prepare the composite by directly combining all of the material from all samples. If it is desired to decrease the total mass of the composite, it is acceptable to combine equal percentages (for example, 75 % or 50 %) of each individual sample, all of which have been divided in the same manner and according to this standard. Determine whether sample masses have been decreased prior to arrival of samples at the laboratory and, if so, by what percentages. Account for that information when making the composite. The samples may be reduced in nominal top size (for example, to pass a 2.36 mm [No. 8] sieve) to facilitate the compositing process. Adhere to the minimum masses required in Table 1. Observe the precautions of Practice D7430 and Test Method D3302/D3302M, as well as those found in this standard, to guard against unaccounted-for changes in moisture. It is advisable to mix the composite sample thoroughly, but without altering the moisture content, before reducing or dividing to smaller quantities.

10.3 If two or more samples have been collected by multiple mechanical coal sampling systems, or if manual sampling practices have been used and only additive parameters are required for analysis, the individual samples can be tested separately and a ton-weighted mathematical average performed, or a composite sample may be prepared. However, if testing is to be performed for non-additive parameters, a composite sample must be prepared. Either 250 μm [No. 60] sieve size samples or samples with larger particle sizes may be used. Minimize the chance for unmeasured changes in moisture by working quickly while samples are exposed to the atmosphere during the compositing process.

10.3.1 If a 250 μm [No. 60] sieve size composite is needed, proceed as follows.

10.3.1.1 Determine the residual moisture content of each 250 μm [No. 60] sieve size sample according to Test Methods D3173/D3173M, D3302/D3302M, or D7582. Designate the results $R_1, R_2, R_3 \dots R_n$.

10.3.1.2 Ascertain the mass of coal in the lot or sub-lot represented by each sample and designate these values $L_1, L_2,$