



# Standard Method of Preparing Coal Samples for Analysis<sup>1</sup>

This standard is issued under the fixed designation D 2013; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This method<sup>2</sup> covers the reduction and division of gross or divided samples, collected in accordance with Test Methods D 2234, 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 weights of 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 procedure of Annex A1.2 to give a sample preparation and analysis variance which is no more than 20 % of the total allowable variance.

1.3 Two methods are given for preparing the analysis sample for making the moisture determinations:

1.3.1 *Referee Method*—This method shall be used where the possibility of unaccounted changes in moisture content during the reduction and division of the gross or divided sample must be held to a minimum. It is intended to be used for evaluation of nonreferee methods and for testing of equipment. Only under certain conditions will this referee method be directly applicable to routine test programs.

1.3.2 *Nonreferee Method*—This method may be used for routine work.

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:

D 197 Test Method for Sampling and Fineness Test of Pulverized Coal<sup>3</sup>

<sup>1</sup> This method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.23 on Sampling.

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<sup>2</sup> For more detailed explanation of this method see Keller, G. E., "Determination of Quantities Needed in Coal Sample Preparation and Analysis," *Transactions*, Vol 232, 1965, pp. 218–226.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.06.

D 410 Method for Sieve Analysis of Coal<sup>4</sup>

D 431 Method for Designating the Size of Coal from its Sieve Analysis<sup>4</sup>

D 2234 Test Methods for Collection of a Gross Sample of Coal<sup>3</sup>

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke<sup>3</sup>

D 3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal<sup>3</sup>

D 3302 Test Method for Total Moisture in Coal<sup>3</sup>

E 11 Specification for Wire-Cloth Sieves for Testing Purposes<sup>5</sup>

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods<sup>5</sup>

## 3. Significance and Use

3.1 This method is intended to provide an analysis sample of coal from a gross or divided sample that has been collected in accordance with Test Methods D 2234. In addition, a method to determine the percent air dried moisture loss of the sample is provided. The analysis sample can be used to determine the value of the coal represented, its ability to meet specifications, its environmental impact, as well as for other purposes.

## 4. Definitions of Terms Specific to This Standard

4.1 *air drying*—a process of partially drying coal to bring moisture near to equilibrium with the atmosphere in the room in which further reduction and division of the sample is to take place.

4.2 *analysis sample*—final subsample prepared from the original gross or divided sample but reduced to 100 % through No. 60 (250- $\mu$ m) sieve and divided to not less than 50 g.

4.3 *bias (systematic error)*—an error that is consistently negative or consistently positive. The mean of errors resulting from a series of observations which does not tend towards zero.

4.4 *C test*—a standard statistical test for homogeneity of variance.<sup>6</sup>

4.5 *divided sample*—a sample that has been reduced in quantity.

4.6 *gross sample*—a sample representing one lot of coal and

<sup>4</sup> *Discontinued; see 1988 Annual Book of ASTM Standards*, Vol 05.05.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>6</sup> Details appear in standard texts. A good text for this purpose is Grubbs, F. E., "An Introduction to Some Precision and Accuracy of Measurement Problems," *JTEVA*, Vol 10, No. 4, July 1982, pp 133–143.

composed of a number of increments on which neither reduction nor division has been performed.

4.7 *laboratory sample*—the sample, not less than the permissible weight given in Table 1, delivered to the laboratory for further preparation and analysis.

4.8 *precision*—a term used to indicate the capability of a person, an instrument, or a method to obtain repeatable results; specifically, a measure of the chance error as expressed by the variance, the standard error, or a multiple of the standard error (see Practice E 177).

4.9 *representative sample*—a sample collected in such a manner that every particle in the lot to be sampled is equally represented in the gross sample.

4.10 *riffle*—a hand-feed sample divider device that divides the sample into two parts of approximately the same weight.

4.11 *sample division*—the process whereby a sample is reduced in weight without significant change in particle size.

4.12 *sample preparation*—the process that may include air drying, crushing, division, and mixing of a gross sample for the purpose of obtaining an unbiased analysis sample.

4.13 *sample reduction*—the process whereby a sample is reduced in particle size by crushing or grinding without significant change.

4.14 *significant loss*—any loss that introduces a bias in final results that is of appreciable economic importance to concerned parties.

4.15 *size consist*—the particle size distribution of a coal.

4.16 *standard deviation*—the square root of the variance.

4.17 *subsample*—a sample taken from another sample.

4.18 *systematic error* (see *bias*, 4.3).

4.19 *top size*—the opening of the smallest screen in the series upon which is retained less than 5 % of the sample (see Method D 431).

4.20 *unbiased sample (representative sample)*—a sample free of bias.

5. Summary of Method

5.1 Three processes of sample division are covered in this method as follows:

5.1.1 *Procedure A*—Riffles are used for division of the sample and mechanical crushing equipment for the reduction of the sample.

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

5.1.3 *Combined Procedure A and B*—The two procedures may be combined at any stage of the preparation procedure.

5.2 These procedures include methods to be used whenever

residual or total moisture or both are to be determined or whenever other laboratory analyses or tests are to be made.

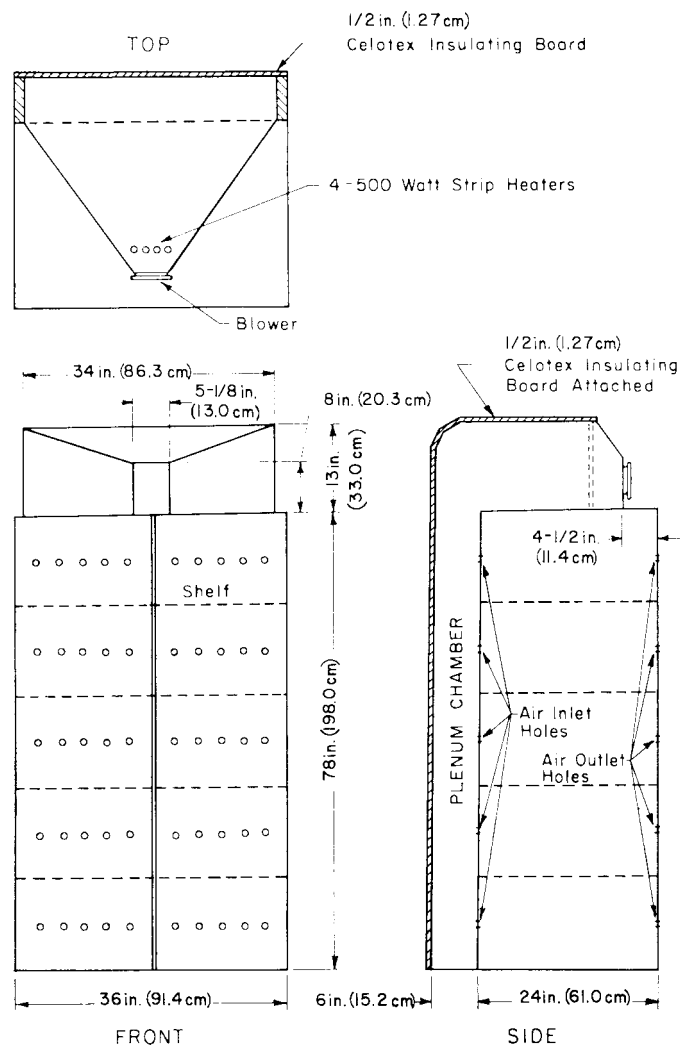
5.3 Preparation of gross or divided samples for analyses or tests consists of air drying (where necessary), particle size reduction, mixing, and dividing the gross or divided sample in stages to a small analysis sample representative of the original gross sample.

6. Apparatus

6.1 *Air Drying*—The following apparatus may be used:

6.1.1 *Air-Drying Oven*—A device for passing slightly heated air over the sample. The oven shall be capable of maintaining a temperature of 10 to 15°C (18 to 27°F) above room temperature with a maximum oven temperature of 40°C (104°F) unless ambient temperature is above 40°C (104°F), in which case, ambient temperature shall be used. In case of easily oxidized coals, the temperature shall not be over 10°C (18°F) above room temperature. Air changes shall be at the rate of 1 to 4/min. A typical oven is shown in Fig. 1.

6.1.2 *Drying Floor*—A smooth clean floor in a room free of dust and excessive air currents.



NOTE 1—Dimensions are typical but required.  
FIG. 1 Air-Drying Oven

TABLE 1 Preparation of Laboratory Sample

Crush to Pass at Least 95 % Through Sieve	Divide to a Minimum Weight of, g <sup>a</sup>	
	Group A	Group B
No. 4 (4.75-mm)	2000	4000
No. 8 (2.36-mm)	500	1000
No. 20 (850 μm)	250	500
No. 60 (250 μm) (100 % through)	50	50

<sup>a</sup>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.

6.1.3 *Drying Pans*—Noncorroding metal pans of sufficient size so that the sample may be spread to a depth of not more than 25 mm (1.0 in.) with sides not more than 38 mm (1.5 in.) high.

6.1.4 *Scale-Gross Sample*—A scale of sufficient capacity and sensitive to 0.023 kg (0.05 lb) in 45.46 kg (100 lb).

6.1.5 *Balance-Laboratory Sample*—A balance of sufficient capacity to weigh the sample and container with a sensitivity of 0.5 g in 1000 g.

6.2 *Crushers or Grinders*—Jaw, cone, or rotary crusher, hammer mill, or other suitable crusher to reduce the sample to pass the sieve designated in Table 1. Hard-steel or chilled-iron plate with tamper, sledge, or hand bar for preliminary crushing of any large lumps in the sample before feeding into the crusher.

6.3 *Pulverizer or Mill*—For final reduction of laboratory sample to pass the No. 60 (250- $\mu$ m) sieve, the following equipment may be used:

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

6.3.2 *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 appreciably increase the ash content of the sample.

6.4 *Bucking Board (Chrome Steel) or Mortar (Agate or Equivalent) and Pestle*—Only for reducing the small fraction of sample, not passing a No. 60 (250- $\mu$ m) sieve after pulverization.

#### 6.5 *Sample Dividers:*

6.5.1 *Mechanical*—A mechanical sample divider using a reciprocating or rotating cutter, a rotating hopper and spout, a rotating slotted cone, or other acceptable devices for dividing the sample. Typical mechanical sample dividers are shown in Fig. 2. These illustrate four designs but others may be available.

6.5.2 *Riffles*—A manual sample divider that splits the coal stream into a number of alternate elements. Riffle divisions should be at least three times the top size of coal being divided. Typical riffler is shown in Fig. 3. It is preferable that feed chutes and enclosed riffles be used. The slope of feed chutes and riffles must be at least 60°.

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

6.5.2.2 *Feed Chute*—A feed chute shall be used as shown in Fig. 3. The discharge opening of the feed chute shall be the same width as the riffle opening.

6.6 *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.

6.7 *Sieves*—A set of sieves whose dimensions are in accordance with Specification E 11, of the following sizes, with cover and receiver:

No.	Size
4	4.75 mm
8	2.36 mm
20	850 $\mu$ m
60	250 $\mu$ m

6.8 *Laboratory Sample Containers*—Heavy vapor-impervious bags, properly sealed, or noncorroding cans such as those with an airtight, friction top or screw top 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 shall be done by trained and experienced personnel. 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, the number and nature of the analysis and tests should be considered. A separate 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.

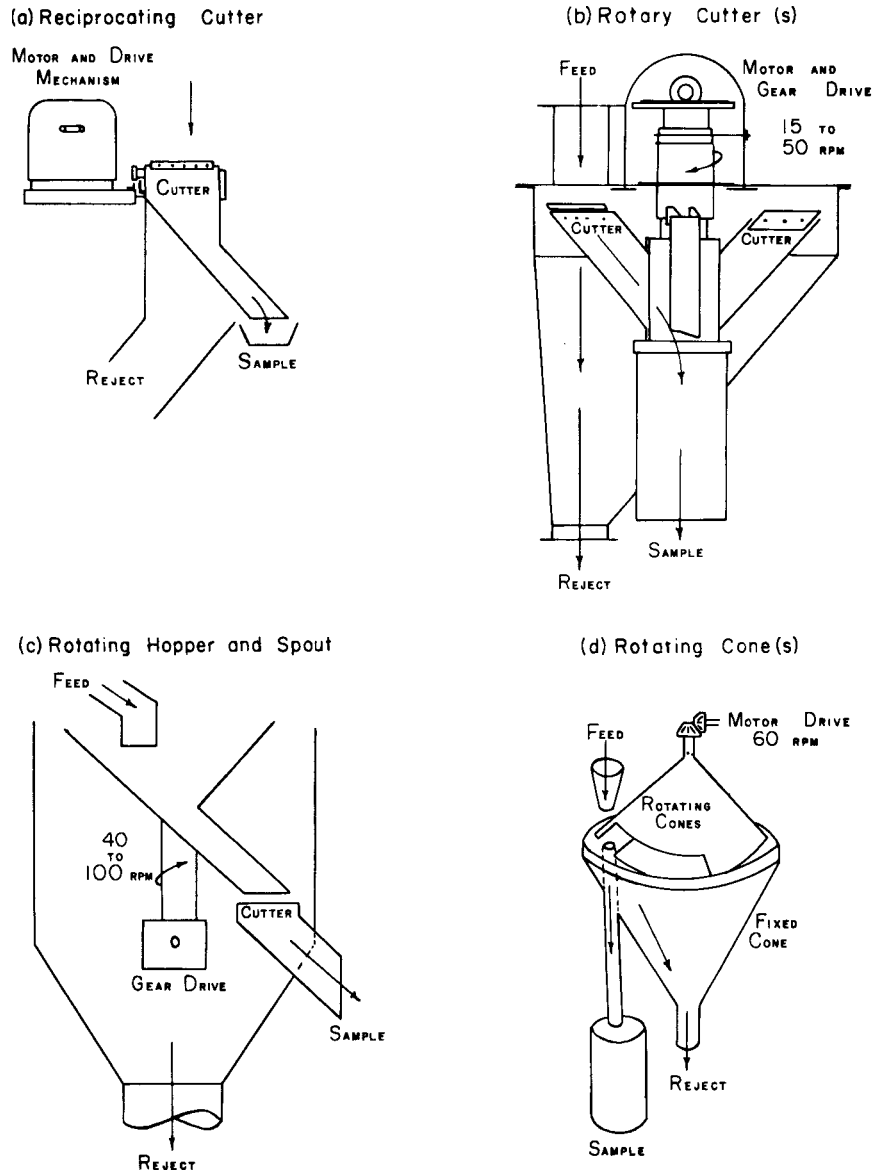
7.3 Since most coals oxidize on exposure to air, the air-drying procedure should not be prolonged past the time necessary to bring its moisture to equilibrium with the air in the room in which further reduction and division are to be made. The sample shall be allowed to attain room temperature before weighing and further reduction.

7.4 In collecting, 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.

7.5 While awaiting preparation, the uncrushed gross or divided sample shall be protected from moisture change caused by exposure to rain, snow, wind, and sun, on contact with absorbent materials.

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

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



(a) *Reciprocating Cutter*—Fig. 2(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. 2(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. 2(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. 2 Mechanical Sample Dividers**

**8. Sieve Tests**

8.1 The errors of sample division are sensitive to the top size (see 4.19) and, therefore, it is important to make a periodic sieve test of the product of the sample crusher. Sieve tests (see 6.7) shall be made and reported in accordance with Method D 410, except when more than 50 % passes the No. 8 (2.36-mm) sieve. Sieve tests on the portions passing the No. 8 (2.36-mm) sieve shall be made in accordance with Test Method D 197.

**9. Procedure**

9.1 *Weights*—The minimum allowable weight of the sample at any stage depends on the size consist, the variability of the

constituent sought, and the degree of precision desired (Table 1).

**9.2 Air Drying:**

9.2.1 *Gross or Divided Samples*—Weigh and spread the referee moisture sample, or a sample too wet to crush without significant loss of moisture, in pans, if an air-drying oven is available, or on a drying floor to a depth of not more than twice the top size of the coal. The coal may be stirred, without loss of coal particles, to speed up air drying. Continue air drying until the loss in weight of the total gross or divided sample is not more than 0.1 %/h. Avoid excessive drying time.

9.2.2 *Laboratory Samples*—Weigh the sample, plus pan,

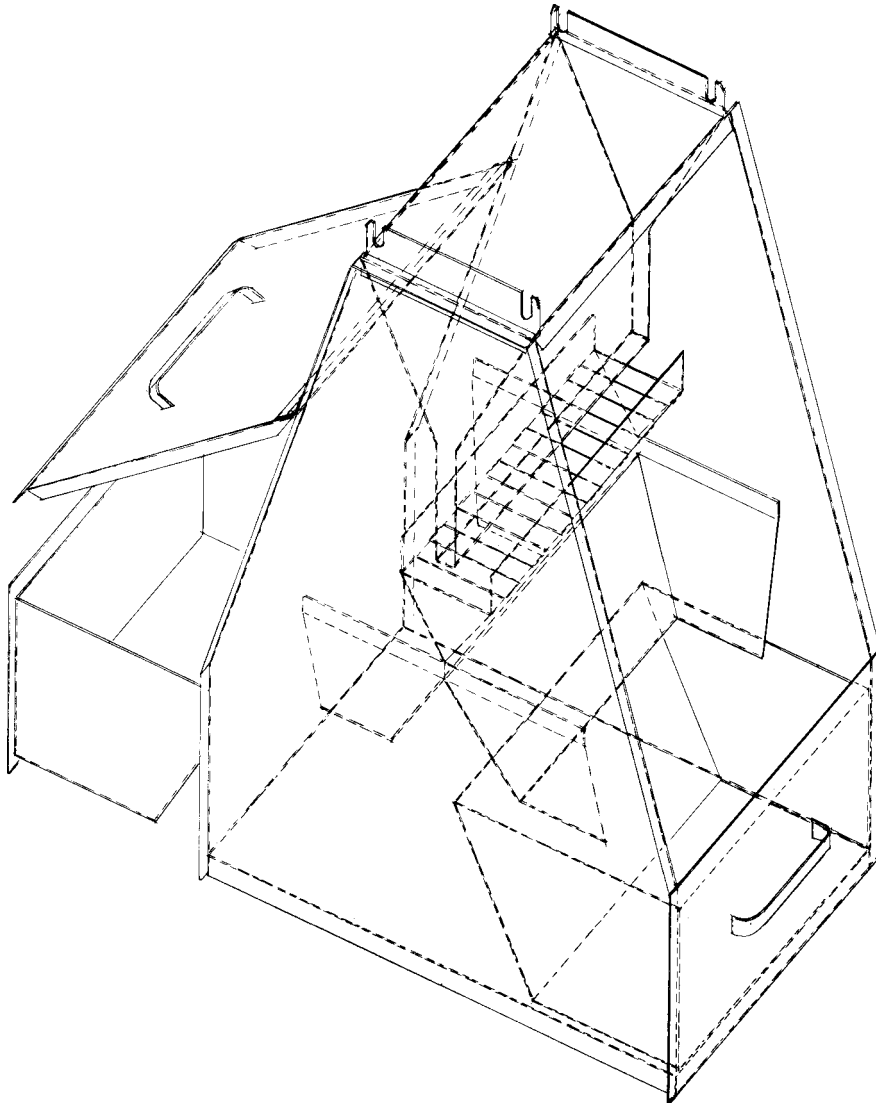


FIG. 3 Sample Divider (Riffle)

plus container, if one is used. Spread the sample in the pan to a depth not to exceed 25 mm (1.0 in.), place it in the oven along with the container and air dry until the loss in weight is not more than 0.1 %/h. Avoid excessive drying. If an oven is not available, the sample may be air dried in a room free from dust and excessive air currents. Stirring at intervals will lessen air-drying time.

9.3 Reduction and Division:

9.3.1 Samples may require air drying to feed properly through the reduction and dividing equipment.

9.3.2 In the reduction and division of gross or divided samples for which total moisture content is to be determined, the precautions in 7.3-7.7 must be followed.

9.3.3 Procedure A— Manual Riffing:

9.3.3.1 Reduce the gross or divided sample to a top size of No. 4 (4.75-mm) or No. 8 (2.36-mm) sieve taking precautions as outlined in Section 7.

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

9.3.3.3 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. 3 and described in 6.5.2. Pass the coal through the riffle from a feed scoop, feed bucket, or riffle pan having a lip or opening the full width of the riffle. When using any of the above 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, thence into the riffle pans, one half of the sample being collected in a 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.

9.3.3.4 If the initial crushing was only to No. 4 (4.75-mm) sieve size, reduce to No. 8 (2.36-mm) sieve size after dividing to not less than the quantity specified in Table 1 for a No. 4 (4.75-mm) sieve size.

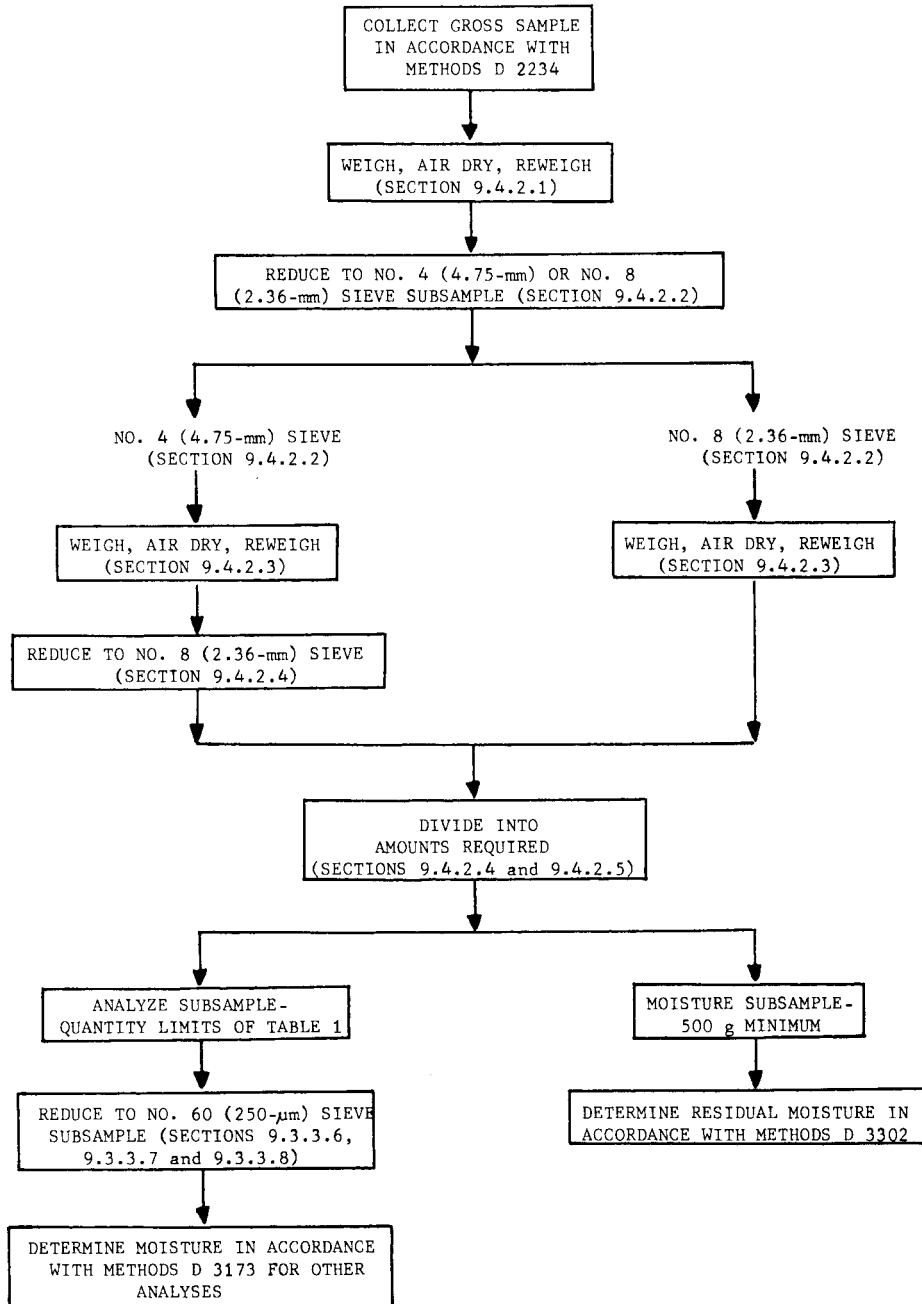


FIG. 4 Gross Sample Preparation for Moisture Determination (Referee Method)

9.3.3.5 After reducing to No. 8 (2.36-mm) sieve size, divide the subsample by riffing to not less than the quantity specified in Table 1 for a No. 8 (2.36-mm) sieve size.

9.3.3.6 With suitable pulverizing equipment (see 6.3), reduce the No. 8 (2.36-mm) sieve size subsample to a No. 60 (250-µm) sieve size. Divide the ground subsample by riffing, using the small riffle (see 6.5.2) until a minimum of 50 g is obtained. Quickly pass the subsample through a No. 60 (250-µm) 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.

9.3.3.7 As an alternative to the procedure of 9.3.3.6 above, the No. 8 (2.36-mm) sieve size subsample may be reduced to

pass 95 % through a No. 20 (850-µm) sieve. Divide this subsample by riffing with the small riffle to not less than the quantity specified in Table 1, and then reduce to No. 60 (250-µm) sieve size as described in 9.3.3.6.

9.3.3.8 Thoroughly mix, preferably by mechanical means, the analysis sample, weighing not less than 50 g, before extracting portions for analysis (see 6.6).

9.3.4 Procedure B— Mechanical Division:

9.3.4.1 Reduce the gross or divided sample in stages and divide by suitable mechanical sample dividers (see 6.5.1) to quantities not less than those shown in Table 1.

9.3.4.2 Mechanical division of the sample consists of automatically collecting a large number of increments of the properly reduced sample. Distribute this large number of

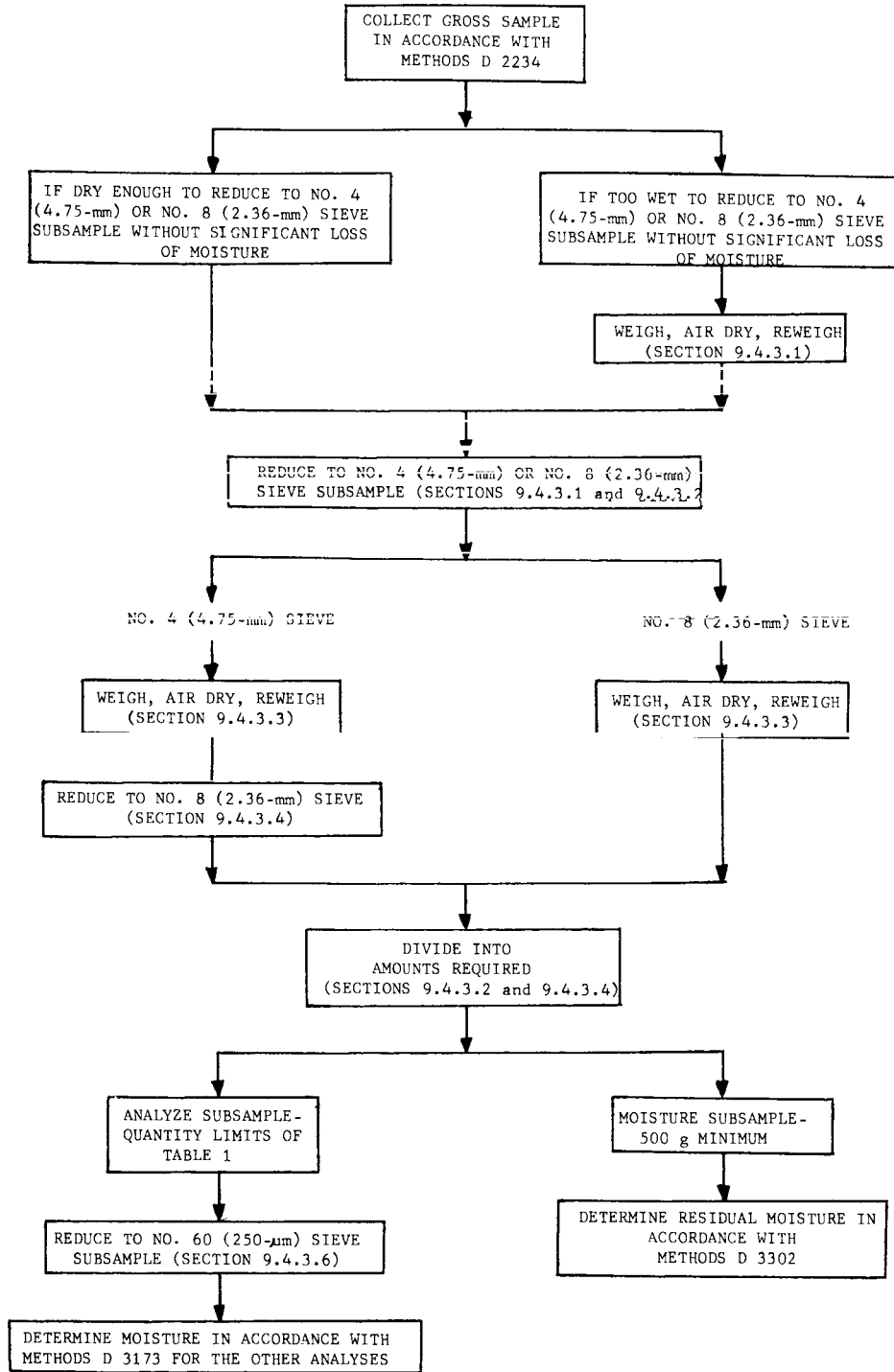


FIG. 5 Gross Sample Preparation for Moisture Determination (Nonreferee Method)

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 where 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.

9.3.4.3 Thoroughly mix the analysis sample, 100 % through No. 60 (250-µm) sieve and weighing not less than 50 g, in

accordance with 9.3.3.8 before extracting portions for analysis.

9.4 Reduction and Division of Moisture Samples:

9.4.1 Two procedures for reduction and division of gross samples for use in moisture determinations are given in 9.4.2, Referee Method, and 9.4.3, Nonreferee Method.

9.4.2 Referee Method—See 1.3.1 and Fig. 4.

9.4.2.1 Before any sample reduction operations are performed, weigh, air dry, and reweigh the entire gross or divided sample in accordance with 9.2.1. The percentage loss is A.

9.4.2.2 Reduce the gross sample to No. 4 (4.75 mm) or No.

8 (2.36 mm) with suitable crushing equipment and divide to quantity limits in Table 1 plus a minimum of 500 g. This is the laboratory sample.

9.4.2.3 Air dry the laboratory sample in accordance with 9.2.2. This air-dry loss is  $A'$ .

9.4.2.4 If the gross or divided sample was reduced originally to No. 4 (4.75 mm), air dry and reduce to No. 8 (2.36 mm). Divide to quantity limits in Table 1 plus 500 g.

9.4.2.5 Divide out the moisture subsample and determine residual moisture in accordance with Test Methods D 3302, Section 2. This is  $R$ .

9.4.2.6 If an analysis sample is required, continue reduction and division in accordance with 9.3.3.6 and 9.3.3.7.

9.4.2.7 Calculate the total moisture,  $M$ , as follows:

$$M' = [R(100 - A')/100] + A'$$

$$M = [M'(100 - A)/100] + A$$

where:

- $M$  = total moisture,
- $M'$  = moisture (laboratory sample),
- $A$  = air-dry loss gross or divided sample,
- $A'$  = air-dry loss (laboratory sample), and
- $R$  = residual moisture.

9.4.3 *Nonreferee Method*—See 1.3.2 and Fig. 5.

9.4.3.1 If the sample is too wet to reduce to No. 4 (4.75 mm) or No. 8 (2.36 mm), air dry the gross or divided sample in accordance with 9.2.1. Complete the preparation and calculations in accordance with 9.4.2.2-9.4.2.7.

9.4.3.2 If the sample is dry enough to reduce to No. 4 (4.75 mm) or No. 8 (2.36 mm), reduce the gross or divided sample with suitable crushing equipment and divide to quantity limits in Table 1 plus 500 g. This is the laboratory sample.

9.4.3.3 *Air-Dry Laboratory Sample*—The percentage air-dry loss is  $A$ .

9.4.3.4 If the sample was reduced to No. 4 (4.75 mm) air dry, reduce to No. 8 (2.36 mm), and divide to quantity limits in Table 1 plus 500 g.

9.4.3.5 Divide out the moisture sample from No. 8 coal and determine the residual moisture in accordance with Test Methods D 3302, Section 2. This is  $R$ .

9.4.3.6 If an analysis sample is required, continue the reduction and division in accordance with 9.3.3.6-9.3.3.8.

9.4.3.7 Calculate the total moisture,  $M$ , as follows:

$$M = [R(100 - A)/100] + A$$

where:

- $M$  = total moisture,
- $A$  = air-dry loss, and
- $R$  = residual moisture.

## 10. Precision and Bias

10.1 The precision of sample preparation (and analysis) can be checked by following Annex A1 and Annex A2. Since this method does not produce a numerical result, determination of bias is not applicable.

## ANNEXES

### (Mandatory Information)

#### A1. METHOD OF CHECKING THE PRECISION OF SAMPLE PREPARATION AND ANALYSIS

##### A1.1 Scope

A1.1.1 This method covers procedures for checking precision of sample preparation and analysis at the various stages. The data obtained from tests using consistent sample preparation and analysis method are used to estimate the random errors in the various stages of sample division and analysis.

A1.1.2 Coals used in each series of tests should be of similar ash content.

##### A1.2 Procedure

A1.2.1 Reduce the gross sample to 95 % through No. 4 (4.75-mm) sieve and divide, using either riffles or mechanical sample dividers, into two equal parts.

A1.2.1.1 Many laboratories are crushing directly to No. 8 (2.36-mm) size instead of to No. 4; but for purpose of test it is usually best to use both No. 4 and 8 sizes since we can assume that crushing directly to No. 8 would give a variance no greater, and probably less, than crushing to No. 4 and then to No. 8. If, however, it is desired to crush directly to No. 8, follow the same procedure as if crushed to No. 4 and then to No. 8.

A1.2.2 Divide each subsample by riffling or mechanically to

no less than weights as outlined in Table 1.

A1.2.2.1 Individual weights should not vary more than  $\pm 20$  % from the weights given in Table 1, and the average of all tests should be within  $\pm 10$  % of the weights.

A1.2.3 Reduce the No. 4 (4.75-mm) sieve laboratory sample 95 % through No. 8 (2.36-mm) sieve and divide, using either riffles or mechanical sample dividers, into two equal parts without discarding. Divide each subsample to no less than the minimum weights as outlined in Table 1.

A1.2.4 Reduce each part of the No. 8 subsample to 100 % through No. 60 (250- $\mu$ m) sieve and divide to no less than 50 g.

A1.2.5 Determine ash in accordance with Test Methods D 3174 in duplicate on each analysis sample.

A1.2.5.1 This test can be used for sulfur, Btu, or other determinations, instead of ash, if desired.

A1.2.5.2 If possible, the duplicate determinations should be made at different times and preferably by different analysts. The purpose of these tests is not to find out how accurate a laboratory can be, but to find out actual variances of preparation and analysis in the normal routine of a laboratory following a prescribed procedure.

A1.2.6 Treat three sets of ten samples each in the above manner.

A1.2.6.1 Make calculations on the first set of ten samples so that the variance for each of the stages may be checked and corrective action, if needed, may be taken.

A1.2.6.2 Continue this cycle of tests until three successive sets of ten samples are satisfactory.

### A1.3 Calculation

A1.3.1 The analysis of variance is based upon the calculations of mean squared differences with the eight determinations for each sample taken in different combinations. Calculate the variances of these combinations:  $VP$ , the variance of the difference between duplicate analyses;  $VQ$ , the variance of the difference between the averages of duplicate analyses; and  $VR$ , the variance of the difference between the average of each four analyses, as follows:

$$VP = (1/4N) \Sigma [(X1 - X2)^2 + (X3 - X4)^2 + (Y1 - Y2)^2 + (Y3 - Y4)^2]$$

where:

$N$  = number of tests.

$X1, X2, X3, X4, Y1, Y2, Y3, Y4$  = individual ash determinations.

$$VQ = \left( \frac{1}{2N} \right) \Sigma \left[ \left( \frac{X1 + X2}{2} - \frac{X3 + X4}{2} \right)^2 + \left( \frac{Y1 + Y2}{2} - \frac{Y3 + Y4}{2} \right)^2 \right]$$

$$VR = (1/N) \Sigma \left[ \left( \frac{X1 + X2 + X3 + X4}{4} - \frac{Y1 + Y2 + Y3 + Y4}{4} \right)^2 \right]$$

A1.3.2 The variances can be resolved further in terms of variance caused by the first stage of sample preparation,  $V1$ ; variance caused by the second stage of sample preparation,  $V2$ ; and the variance of analysis,  $Va$ .

where:

$$Va = \frac{1}{2} VP,$$

$$V2 = \frac{1}{2} VQ - \frac{1}{4} VP, \text{ and}$$

$$V1 = \frac{1}{2} VR - \frac{1}{4} VQ.$$

A1.3.3 The total variance of sample preparation and analysis,  $S_{da}^2$ , is given by the equation:

$$S_{da}^2 = Va + V2 + V1$$

A1.3.4 The calculations of the variances of sample preparation are illustrated in Table A1.1.

**TABLE A1.1 Illustrations of the Calculation of the Variances<sup>A</sup> of Sample Preparation at the Various Stages and Analysis**

Test No.	X1	X2	Difference	Difference <sup>2</sup>	X3	X4	Difference	Difference <sup>2</sup>
1	12.13	12.10	0.03	0.0009	12.03	12.05	-0.02	0.0004
2	10.67	10.73	-0.06	0.0036	10.69	10.78	-0.09	0.0081
3	10.93	11.10	-0.17	0.0289	11.36	11.45	-0.09	0.0081
4	12.05	12.02	0.03	0.0009	12.17	12.23	-0.06	0.0036
5	12.74	12.70	0.04	0.0016	12.71	12.76	-0.05	0.0025
6	12.47	12.30	0.17	0.0289	12.21	12.14	0.07	0.0049
7	11.94	11.99	-0.05	0.0025	12.08	12.17	-0.09	0.0081
8	12.52	12.63	-0.11	0.0121	12.76	12.82	-0.06	0.0036
9	12.01	12.05	-0.04	0.0016	11.94	11.77	0.17	0.0289
10	10.96	10.88	0.08	0.0064	11.37	11.40	-0.03	0.0009
Total	118.42	118.50		0.0874	119.32	119.57		0.0691
Average	11.84	11.85			11.93	11.96		

Test No.	Y1	Y2	Difference	Difference <sup>2</sup>	Y3	Y4	Difference	Difference <sup>2</sup>
1	12.00	12.01	-0.01	0.0001	12.00	12.00	0.00	0.0000
2	10.53	10.65	-0.12	0.0144	10.60	10.62	-0.02	0.0004
3	11.37	11.47	-0.10	0.0100	11.22	11.35	-0.13	0.0169
4	12.13	12.10	0.03	0.0009	12.01	12.04	-0.03	0.0009
5	12.60	12.60	0.00	0.0000	12.51	12.40	0.11	0.0121
6	12.09	12.15	-0.06	0.0036	12.18	12.20	-0.02	0.0004
7	11.93	11.87	0.06	0.0036	11.71	11.73	-0.02	0.0004
8	12.57	12.57	0.00	0.0000	12.58	12.61	-0.03	0.0009
9	11.81	11.88	-0.07	0.0049	11.70	11.84	-0.14	0.0196
10	11.57	11.48	0.09	0.0081	11.54	11.36	0.18	0.0324
Total	118.60	118.78		0.0456	118.05	118.15		0.0840
Average	11.86	11.88			11.81	11.82		

Test No.	X(1 + 2)/2	X(3 + 4)/2	Difference	Difference <sup>2</sup>	Y(1 + 2)/2	Y(3 + 4)/2	Difference	Difference <sup>2</sup>
1	12.11	12.04	0.07	0.0056	12.00	12.00	0.00	0.0000
2	10.70	10.73	-0.03	0.0012	10.59	10.61	-0.02	0.0004
3	11.01	11.40	-0.39	0.1521	11.42	11.28	0.13	0.0182
4	12.03	12.20	-0.16	0.0272	12.11	12.02	0.09	0.0081
5	12.72	12.73	-0.01	0.0002	12.60	12.45	0.14	0.0210
6	12.38	12.17	0.21	0.0441	12.12	12.19	-0.07	0.0049
7	11.96	12.12	-0.16	0.0256	11.90	11.72	0.18	0.0324
8	12.57	12.79	-0.21	0.0462	12.57	12.59	-0.02	0.0006
9	12.03	11.85	0.17	0.0306	11.84	11.77	0.07	0.0056
10	10.92	11.38	-0.46	0.2162	11.52	11.45	0.07	0.0056
Total	118.46	119.44		0.5491	118.69	118.10		0.0969
Average	11.85	11.94			11.87	11.81		

Test No.	X(1 + 2 + 3 + 4)/4	Y(1 + 2 + 3 + 4)/4	Difference	Difference <sup>2</sup>
1	12.07	12.00	0.07	0.0056
2	10.71	10.60	0.11	0.0138
3	11.21	11.35	-0.04	0.0203
4	12.11	12.07	0.04	0.0022
5	12.72	12.52	0.20	0.0400
6	12.28	12.15	0.12	0.0156
7	12.04	11.81	0.23	0.0552
8	12.68	12.58	0.10	0.0100
9	11.94	11.80	0.13	0.0182
10	11.15	11.48	-0.33	0.1122
Total	118.95	118.39		0.2932
Average	11.90	11.84		

$$VP = \frac{1}{40} (0.0874 + 0.0691 + 0.0456 + 0.0840) = 0.0071$$

$$VQ = \frac{1}{20} (0.5491 + 0.0969) = 0.0323$$

$$VR = \frac{1}{10} (0.2932) = 0.0293$$

Then:

$$Va = \frac{1}{2} (0.0071) = 0.0035$$

$$V_2 = \frac{1}{2} (0.0323) - \frac{1}{4} (0.0071) = 0.0144$$

$$V_1 = \frac{1}{2} (0.0293) - \frac{1}{4} (0.0323) = 0.0066$$

$$S_{da}^2 = 0.0035 + 0.0144 + 0.0066 = 0.0245$$

<sup>A</sup>This table contains data taken from a computer printout with rounding errors that are not involved in the overall calculation. Data taken at intermediate steps are not consistent within limits of these rounding errors. Thus, the difference 0.07<sup>2</sup> shows a result of 0.0056 which is correct when all places are carried in the calculation.

## A2. METHOD FOR DETERMINING THE OVER-ALL VARIANCE OF DIVISION AND ANALYSIS<sup>4</sup>

### A2.1 Scope

A2.1.1 Legitimate estimates of the variance of division and analysis,  $S_{da}^2$ , can only be made using data obtained from tests that were run using consistent division and analysis methods. Coals used in these variance tests should be of similar ash content. Any gross change in the division and analysis methods or in the ash characteristics of the test coal will nullify the test results.

### A2.2 Procedure

A2.2.1 The following four-step method uses the regular gross or divided samples obtained from normal sampling operations:

A2.2.1.1 Crush the gross sample to the same mesh as that normally obtained when preparing the gross sample for processing,

A2.2.1.2 Divide the sample into four equal parts, according to the normal routine laboratory procedure,

A2.2.1.3 Reduce the four subsamples to laboratory analysis samples, and

A2.2.1.4 Analyze each analysis sample for dry ash content.

A2.2.2 Calculate the variance of division and analysis for each gross or divided sample from the “within set sums of squares” for the replicate determinations as follows:

$$S_{da}^2 = [\sum x^2 - (\sum x)^2/4]/3$$

where:

$S_{da}^2$  = variance of division and analysis,

$x^2$  = sum of the squares of the four ash results, and

$(\sum x)^2$  = sum of the ash results, quantity squared.

A2.2.3 Make progressive checks as the work is carried out by using the data in groups of 5. In any group of 5 estimates of  $S_{da}^2$  based on 4 subsamples for each estimate, the ratio of the largest estimate to the average of the group should not exceed 2.99, in 19 out of 20 cases. Investigate values in excess of this ratio before proceeding with the test. In addition, after completing 30 sets, by groups of 5, the ratio of the largest group average to the overall average should not exceed 1.88 in 19 cases out of 20. If these criteria are met, the variance of division and analysis may be taken as the overall average  $S_{da}^2$  of the 30 sets of data. If these criteria are not met, follow the procedure described in Method D 2013 for the necessary information to improve techniques of division and analysis.

A2.2.4 *Example*—A complete example illustrating the procedure for determining the variance of division and analysis is given in Table A2.1. In this example, gross sample No. 24, the highest individual ash sample in the group (19.28 % ash), has an unusually high variance of division and analysis. The behavior of samples 21 to 30 indicates that trouble can be expected when the ash exceeds 15 % (see Table A2.1).

**TABLE A2.1 Determination of Variance of Division and Analysis—Use of Four Analysis Samples for Each Gross Sample**

NOTE 1—10 % ash was subtracted from each of the ash results listed to simplify the calculations.

Gross Sample Number	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)
	Analysis Samples				$\Sigma x$	$\Sigma x^2$	$(\Sigma x)^2/4$	(6)–(7)	$(8)/3 = S_{da}^2$	Average Sets of 5 $S_{da}^2$	$C_i^A$	$C_i^B$
1	1.22	1.37	1.56	1.71	5.86	8.7230	8.5849	0.1381	0.0460		1.62	
2	1.29	1.17	1.70	1.57	5.73	8.3879	8.2082	0.1797	0.0599		2.11	
3	1.56	1.66	1.58	1.64	6.44	10.3752	10.3684	0.0068	0.0023		0.08	
4	5.63	5.57	5.93	5.52	22.65	128.3571	128.2556	0.0015	0.0005		0.02	
5	3.90	3.87	3.58	3.56	14.91	55.6769	55.5770	0.0999	0.0333		1.17	
Average	...	...	...	...	12.78	...	...	...	...	0.0284	...	0.61
6	0.64	0.42	0.80	0.73	2.59	1.7589	1.6770	0.0819	0.0273		1.12	
7	2.47	2.44	2.74	2.68	10.33	26.7445	26.6772	0.0673	0.0227		0.93	
8	3.70	3.53	3.43	3.43	14.09	49.6807	49.6320	0.0487	0.0162		0.66	
9	3.59	3.73	4.13	3.80	15.25	58.2979	58.1406	0.1573	0.0524		2.15	
10	2.14	2.17	2.25	2.11	8.67	18.8031	18.7922	0.0109	0.0036		0.15	
Average	...	...	...	...	12.55	...	...	...	...	0.0244	...	0.52
11	5.71	5.61	5.61	5.71	22.64	128.1524	128.1424	0.0100	0.0033		0.09	
12	3.21	3.40	2.86	2.90	12.37	38.4537	38.2542	0.1995	0.0665		1.87	
13	4.99	4.80	5.51	4.93	20.23	102.6051	102.3132	0.2919	0.0973		2.74	
14	3.26	3.15	3.17	3.09	12.67	40.1471	40.1322	0.0149	0.0050		0.14	
15	3.48	3.65	3.59	3.53	14.25	50.7819	50.7656	0.0163	0.0054		0.15	
Average	...	...	...	...	14.11	...	...	...	...	0.0355	...	0.76
16	2.89	2.84	2.85	2.89	11.47	32.8923	32.8902	0.0021	0.0007		0.02	
17	2.35	2.48	2.90	2.71	10.44	27.4270	27.2484	0.1786	0.0595		1.86	
18	4.23	3.92	4.13	4.05	16.33	66.7187	66.6672	0.0515	0.0172		0.54	
19	5.46	5.13	5.13	5.38	21.10	111.3898	111.3025	0.0873	0.0291		0.91	
20	3.15	2.98	3.42	3.47	13.02	42.5402	42.3801	0.1601	0.0534		1.67	
Average	...	...	...	...	13.62	...	...	...	...	0.0320	...	0.69
21	2.88	2.81	2.80	2.59	11.08	30.7386	30.6916	0.0470	0.0157		0.17	
22	4.94	4.32	4.40	4.39	18.05	81.6981	81.4506	0.2945	0.0982		1.05	
23	4.04	4.28	4.47	4.48	17.27	74.6913	74.5632	0.1281	0.0427		0.46	
24	8.38	8.28	8.93	9.28	34.87	304.6461	303.9792	0.6669	0.2223		2.39	
25	6.93	6.97	6.37	6.54	26.81	179.9543	179.6940	0.2603	0.0868		0.93	
Average	...	...	...	...	15.40	...	...	...	...	0.0931	...	2.00 <sup>C</sup>
26	4.52	4.27	3.66	4.07	16.52	68.6238	68.2276	0.3962	0.1321		2.02	
27	4.53	4.46	4.54	4.65	18.18	82.6466	82.6281	0.0185	0.0062		0.09	
28	2.18	2.42	2.45	2.31	9.36	21.9474	21.9024	0.0450	0.0150		0.23	
29	8.84	9.21	8.69	8.55	35.29	311.5883	311.3460	0.2423	0.0808		1.24	
30	5.03	4.73	5.47	5.11	20.34	103.7068	103.4289	0.2779	0.0926		1.42	
Average	...	...	...	...	14.98	...	...	...	...	0.0653	...	1.40
Overall average $S_{da}^2$	...	...	...	...	...	...	...	...	...	0.0465	...	...

<sup>A</sup> $C_i^m$  for individuals in subgroup. Divide individual  $S_{da}^2$  values (Column 9) by average  $S_{da}^2$  (Column 10). Results should be below 2.99 in 19 cases out of 20.

<sup>B</sup> $C_i^m$  for subgroup averages. Divide average  $S_{da}^2$  (Column 10) by overall averages  $S_{da}^2$ . Result should be below 1.88 in 19 cases out of 20.

<sup>C</sup>Above limit of 1.88.

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