



# Standard Test Method for Determining the Washability Characteristics of Coal<sup>1</sup>

This standard is issued under the fixed designation D 4371; 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.

## INTRODUCTION

The American Society for Testing and Materials has recognized the need for a standard procedure to conduct washability analyses that will serve as an aid to technical communication among individuals and organizations that supply, purchase, and utilize coal. This test method standardizes procedures utilized for performing washability analyses, the data from which can be used for interpreting preparation plant efficiency, for determining preparation plant design, and for determining the potential recovery and quality of coal reserves.

### 1. Scope

1.1 This test method describes procedures for determining the washability characteristics of coarse-coal fractions and fine-coal fractions. Each sample being tested can have more than one coarse-coal size fraction and more than one fine-coal size fraction (see Annex A1). Each such size fraction has an upper size limit and a lower size limit, neither of which overlaps with the next larger nor smaller size fraction. (See Test Method D 4749.)

1.2 While, in general, this test method has useful application in determining the washability characteristics of low-rank coals, in the strictest sense, it is not necessarily the most technically correct test method to determine washability characteristics of low-rank coals because of problems relative to the loss of moisture through drying during sample preparation and analysis. Methods that are directly applicable to low-rank coals are to be developed.

1.3 The values stated in SI units are to be regarded as the standard. The values shown in parentheses are for information only.

1.4 *This standard does not purport to address 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.* For specific hazard statements, see Section 8 and Appendix X1.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 2013 Method of Preparing Coal Samples for Analysis<sup>2</sup>
- D 2234 Practice for Collection of a Gross Sample of Coal<sup>2</sup>
- D 4749 Test Method for Performing Sieve Analysis of Coal and Designating Coal Size<sup>2</sup>
- D 4915 Guide for Manual Sampling of Coal from Tops of Railroad Cars<sup>2</sup>
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes<sup>3</sup>
- E 100 Specification for ASTM Hydrometers<sup>4</sup>
- E 126 Test Method for Inspection and Verification of Hydrometers<sup>4</sup>
- E 323 Specification for Perforated-Plate Sieves for Testing Purposes<sup>3</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *coal washability*—the determination of the theoretical limits for the removal of mineral impurities from coal by beneficiation processes that rely on specific gravity separations.

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *washability analysis of coal*—the analysis of the specific gravity distribution of chemical and physical characteristics of coal.

3.2.1.1 *Discussion*—In this test method, the specific gravity fractions are obtained by subjecting the material being studied to a series of solutions, each with a discrete specific gravity, that cover the range of specific gravities in question. In the case of the washability analysis of coal, these solutions are obtained by the mixing of various organic liquids that are relatively inert toward the majority of coal types. The distribution, as determined by the analysis, is affected by the physical condition of

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.07 on Physical Characteristics of Coal.

Current edition approved Feb. 22, 1991. Published May 1991. Originally published as D 4371 – 84. Last previous edition D 4371 – 84.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.05.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.03.

the sample subjected to the washability analysis, for example, the moisture content and the size content of the material.

3.2.2 *coarse coal*—that portion of a coal sample being subjected to a washability study that is larger than a specific predetermined particle size, generally between 2.36 mm (No. 8 USA Standard Sieve Series) and 9.5 mm (3/8 in.) round in diameter.

3.2.2.1 *Discussion*—This same particle size breakpoint should then be used in subsequent washability studies of the same material sampled from the same location for the same application. This breakpoint is determined by the analyst or the person designing the test procedure as the point that best suits the application. This coarse-coal fraction may be further sieved (generally by dry-sieving) to produce additional size fractions, each of which is processed through the desired specific gravity solutions.

3.2.3 *fine coal*—that portion of a coal sample being subjected to a washability study that is smaller than the predetermined particle size generally between 2.36 mm (No. 8 USA Standard Sieve Series) and 9.5 mm (3/8 in.) round in diameter, which is specified in 3.2.2.

3.2.3.1 *Discussion*—This same particle size breakpoint should then be used in subsequent washability studies of the same material sampled from the same location for the same application. This breakpoint is determined by the analyst or the person designing the test procedure as the point which best suits the application. This fine-coal fraction may be further sieved (generally by wet-sieving) to produce additional size fractions, each of which is processed through the desired specific gravity solutions.

3.2.4 *float/sink*—a reference to the physical action that particles undergo when immersed in a liquid of a predetermined specific gravity.

3.2.4.1 *Discussion*—A series of float/sink tests is considered as being synonymous with a washability analysis. A float fraction or float material is the material or the specific gravity fraction that floats in a certain solution of specific gravity liquids. A sink fraction or sink material is the material or the specific gravity fraction that sinks in a certain solution of specific gravity liquids.

## 4. Summary of Test Method

4.1 *Procedure A*—Describes a washability procedure for coal fractions composed of particles of coal that are larger than a specified, predetermined size, generally between 2.36 mm (No. 8 USA Standard Sieve Series) and 9.5 mm (3/8 in.) round in diameter. These coal fractions will be referred to as coarse-coal fractions.

4.1.1 Each coarse-coal size fraction is independently separated into two distinct specific gravity fractions by placing the sample into a large tank of sufficient capacity, for example, 40 to 80 L (10 to 20 gal), containing a screen basket insert and a solution of organic liquids adjusted to the required specific gravity. The suspension is stirred gently to assure that all particles are allowed to either float or sink. The material that floats on the solution is removed first, followed by the portion of sample that sinks.

4.2 *Procedure B*—Describes a washability procedure for coal fractions composed of particles of coal that are smaller

than the specified, predetermined size chosen in 4.1. These coal fractions will be referred to as fine-coal fractions.

4.2.1 Each fine-coal size fraction is separated into two distinct specific gravity fractions by placing a portion of the fine-coal sample into a float/sink flask of sufficient capacity, for example, 2 to 4 L (0.5 to 1 gal), containing a solution of organic liquids adjusted to the required specific gravity (Note 1, Note 2, Note 3, and Note 4). Extreme care must be exercised to avoid overloading the float/sink flask. This suspension is then stirred to be certain that each individual particle is allowed to float or sink. The material that floats is removed first by carefully inserting a stopper into the neck of the flask and pouring the organic solution and float material from the upper portion of the float/sink flask through a filtering medium that traps the float material. The sink material is then removed and captured in a similar manner.

NOTE 1—A limited number of laboratories are currently using separatory funnels for the separation of fine coal particles having a diameter less than one third the diameter of the stopcock port.

NOTE 2—Some laboratories use centrifugal methods of separating fine-coal density fractions. Centrifugal methods are not a part of this test method. A possible source of information is the Bureau of Mines' Report of Investigations 5741 (1),<sup>5</sup> or the Department of Energy, Pittsburgh Energy Technology Center, Coal Preparation Branch, P.O. Box 10940, Pittsburgh, PA 15236.

NOTE 3—Preparation and adjustment of the organic solutions should be done in an unrestricted container to allow proper mixing of the reagents prior to their introduction into the float/sink flasks.

NOTE 4—It is a general guide that the depth of the particles floating should not exceed three to five times the mean diameter of the particles in the sample. It is suggested to use 200 g, or less.

### 4.3 Preparation of Test Sample, Sieving:

4.3.1 The bulk sample should be spread on an impervious base, preferably under shelter. The sample should be dried and sieved in accordance with Test Method D 4749, using a suitable range of apertures. (Apertures must conform to Specification E 11 or E 323.)

4.3.2 Where it is possible that some fines agglomerate or adhere to larger particles, it is important that wetsieving be used to ensure that fine material reports to the correct particle size fraction. However, under normal circumstances where air-drying has occurred, it should not be necessary to wet-sieve the material larger than 3.15 mm (1/8 in.).

4.3.3 As a practical guide, the presence of minus 75- $\mu$ m (No. 200 USA Standard) material should be minimized, because Brownian movement or molecular interference can be detrimental to proper and efficient separations. Minus 75- $\mu$ m (No. 200 USA Standard) material can then be processed using separate test procedures. (See, for example, the reference in Note 2.)

4.3.4 When required, material which is larger than the desired topsize is broken by hand or machine-crushed according to the topsize required, or, if applicable, by simulating the relevant part of the crushing circuit.

<sup>5</sup> The boldface numbers in parentheses refer to the list of references appended to this standard.

## 5. Significance and Use

5.1 Although the determination of the washability characteristics of coal is not an exact science, this washability test method can be used to investigate the cleaning characteristics of coarse- and fine-coal fractions. However, especially with the fine-coal fractions, this test method may not be applicable for low-rank coals for reasons explained in 1.2.

5.2 The usefulness of this test procedure and the usefulness of the results derived from washability testing are directly related to the care exercised in developing and following the sampling regime while taking into consideration the desired degree of precision. For more exacting work, it will be necessary to know the general washability characteristics of the material in question. This can be done only through pilot testing as described in 9.6 and Annex A3.

5.3 This test method, in conjunction with the advice of experts and with knowledge of the particle size analysis (see Test Method D 4749), is used for the engineering design of coal preparation facilities.

5.4 This test method is also used to assist in process control and to determine the efficiency of operating preparation plants.

5.5 This test method is used in core studies to determine the quality washability characteristics of coal reserves; however, in these instances the typical mass of raw coal from Table 1 may be neither applicable nor practical. For this reason, core diameter should be as large as possible and subdivision of the core section by any means or for any purpose prior to washability testing should be avoided. Care must be exercised in the design of the coring programs, including the consideration of appropriate geostatistics, in order that the potential recovery and quality of the coal reserves, as defined by the washability analysis of the cores, have any practical significance.

## 6. Apparatus

6.1 The suggested apparatus to be used should be as simple as possible and be commensurate with the test objective—the principal criteria being that it should be unaffected by the solutions involved (that is, the organic liquids do not corrode, etch, or otherwise react with the materials from which the apparatus is constructed) and that it should be convenient for use (see X1.3). Types of apparatus that have been found to be

suitable are discussed in the following subsections and are illustrated in Fig. 1, Fig. 2, and Fig. 3.

6.2 *Suitable Float/Sink Container for Coarse-Coal Fractions*—The float/sink apparatus may consist of a wire mesh insert and a holding tank (Fig. 1 and Fig. 2). The wire mesh insert should consist entirely of wire mesh, have solid sides with a wire mesh bottom, or have a solid bottom with wire mesh sides. The wire mesh used must be smaller than one half the size of the finest coal in the fraction. The insert fits inside the holding tank. The insert may have handles attached or may utilize an overhead lifting device to facilitate removal from the tank. A typical laboratory holding tank has approximately 40 to 80-L (10 to 20-gal) capacity. Tank and insert must be designed so as to be compatible.

NOTE 5—It is possible that the geometry of the tank may have some effect on the ease with which the materials are removed from the tank. Square tanks have the following disadvantages: (a) it is difficult to get the wire mesh strainer into the corners to remove all of the float material, and (b) material, especially the sink fraction, may be trapped in the corners while the material is being removed from the tank.

6.3 *Suitable Float/Sink Flask for Fine-Coal Fractions*—The float/sink flasks, that typically have a capacity of 2 to 4 L (0.5 to 1 gal), should be constructed with ground glass connecting joints (see Fig. 3).

### 6.4 Additional Apparatus:

6.4.1 *Balance*, suggested 2000-g capacity, sensitive to  $\pm 0.1$  g, for weighing small samples or fractions.

6.4.2 *Balance*, suggested 0 to 110-kg (0 to 250-lb) capacity, sensitive to  $\pm 45$  g (0.10 lb) for weighing large samples or fractions.

6.4.3 *Wire Mesh Strainers*, with mesh size one half of the size of the smallest particle.

6.4.4 *Vacuum or Pressure Filter, or Filter Funnel* for gravity filtration.

6.4.5 *Drying Oven*, forced air, heatable to 40°C with adequate and proper ventilation.

6.4.6 *Hydrometers*, series within the working range desired, compatible with Specification E 100 and Test Method E 126.

6.4.7 *Hydrometer or Graduated Cylinder* of sufficient size.

6.4.8 *Dispenser*, plastic, squeeze, unaffected by the organic liquids being used.

## 7. Reagents and Materials

7.1 *General Consideration*—Media that have been used for the separation include solutions of organic liquids, aqueous solutions of inorganic salts, and solids in aqueous suspensions. The choice of medium is governed to some extent by the bulk and particle size of the coal being tested, its rank, relative density, and the purpose for which the separation is being performed. Solutions of organic liquids are the preferred medium and it is recommended that aqueous suspensions not be used as the medium for washability analysis.

7.1.1 The most suitable range of relative densities is dependent upon the specific purpose of the test and the characteristics of the specific coal, but would typically include 1.3, 1.4, 1.5, 1.6, 1.7, 1.8, and 2.0 specific gravities with additional gravities if desired.

7.2 *Organic Liquids*—The following organic liquids, shown with their relative densities, are generally used for preparation

**TABLE 1 Typical Coal Mass For Four To Six Specific Gravity Fractions**

Size Fraction	Mass of Material Needed for Washability Analysis of that Size Fraction, kg (lb)
200 mm $\times$ 100 mm (8 in. $\times$ 4 in. round)	2720 (6000)
100 mm $\times$ 50 mm (4 in. $\times$ 2 in. round)	910 (2000)
50 mm $\times$ 25 mm (2 in. $\times$ 1 in. round)	225 (500)
25 mm $\times$ 12.5 mm (1 in. $\times$ ½ in. round)	90 (200)
12.5 mm $\times$ 6.3 mm (½ in. $\times$ ¼ in. round)	25 (50)
6.3 mm $\times$ 2.36 mm (¼ in. $\times$ No. 8 USA Standard)	9 (20)
2.36 mm $\times$ 1.40 mm (No. 8 $\times$ No. 14 USA Standard)	5 (10)
1.40 mm $\times$ 600 $\mu$ m (No. 14 $\times$ No. 30 USA Standard)	2 (5)
600 $\mu$ m $\times$ 300 $\mu$ m (No. 30 $\times$ No. 50 USA Standard)	1 (2)
300 $\mu$ m $\times$ 150 $\mu$ m (No. 50 $\times$ No. 100 USA Standard)	0.5 (1)
150 $\mu$ m $\times$ 75 $\mu$ m (No. 100 $\times$ No. 200 USA Standard)	0.5 (1)

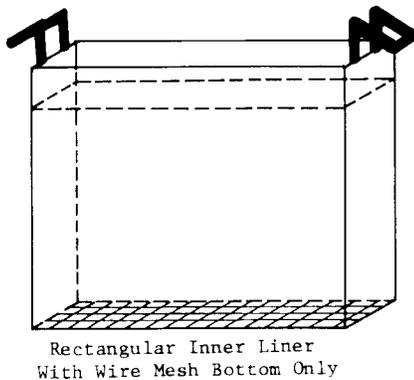
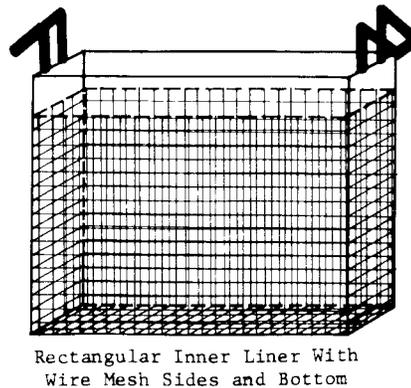
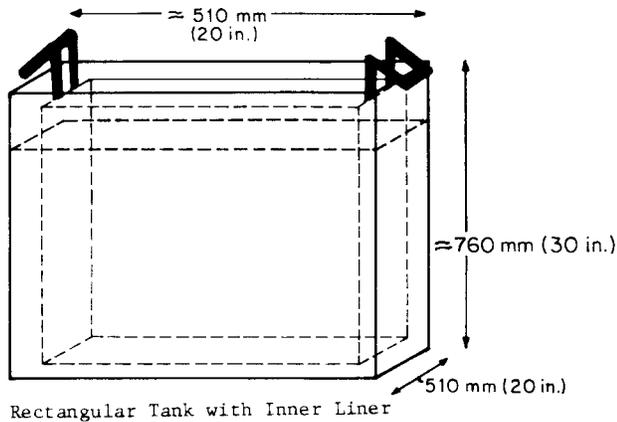


FIG. 1 Suitable Rectangular Float and Sink Apparatus for Separation of Coarse-Coal Fractions

of washability media: petroleum spirit (0.7), white spirit (0.77), naphtha (0.79), toluene (0.86), perchloroethylene (1.6), methylene bromide (2.49), bromoform (2.9), and tetrabromoethane (2.96).

7.2.1 Information on preferability for organic liquids, their volatility, specific gravity, specific gravity measurements, and subsequent treatment for removal is included in Annex A2.

NOTE 6—**Warning:** Some of these organic liquids may be hazardous. See Appendix X1.

## 8. Hazards

8.1 The most commonly used organic solutions produce vapors and must be used only in a well-ventilated or hooded area. In addition to adequate ventilation, it is also prudent to use appropriate safety measures. (See Appendix X1.)

## 9. Sampling and Test Specimens

9.1 Obtaining analytical results that truly represent the material being sampled requires planning and diligence in taking the sample and its storage, handling, and preparation (2, 3, 4, 5, 6, 7, 8, 9). The wide varieties of coal handling facilities, the wide differences in physical constraints on the various sampling locations, and the various uses of coal washability data preclude the publication of detailed recommendations for the sampling of coal for every situation. Sampling and sample preparation, therefore, should be done in accordance with Test Methods D 2234, Practice D 4915 and Method D 2013. To assure that a sufficient amount of material is collected, refer to Table 1.

9.2 The quantity of sample and the degree of accuracy

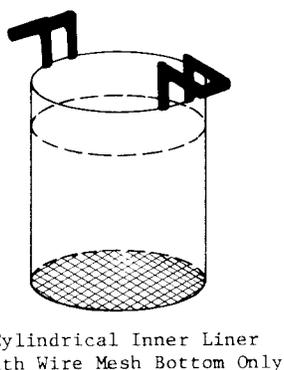
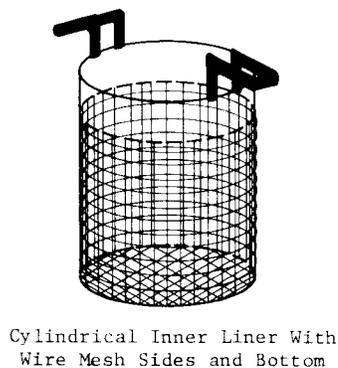
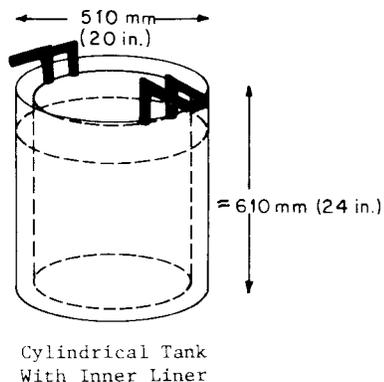
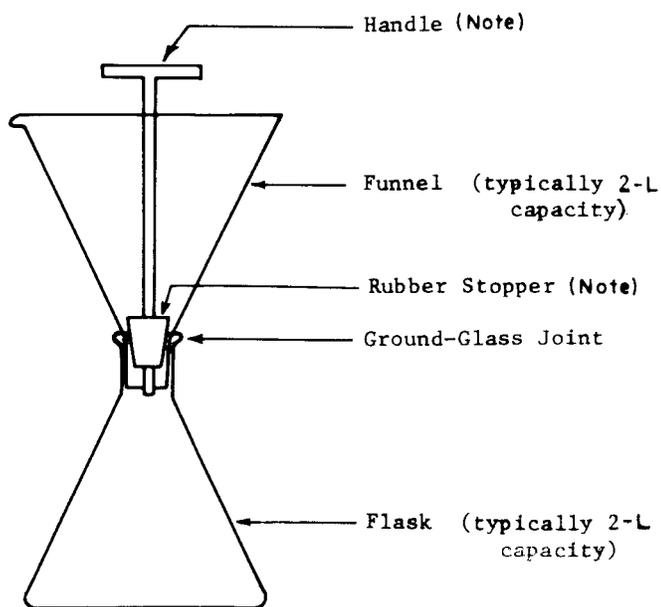


FIG. 2 Suitable Cylindrical Float and Sink Apparatus for Separation of Coarse-Coal Fractions



NOTE 1—Handle and rubber stopper are put in place as shown only when the float/sink flasks are to be disassembled for recovering the float and sink fractions.

FIG. 3 Suitable Float and Sink Apparatus for Separation of Fine-Coal Fractions

desired in a float and sink test varies according to the purpose for which the test is being performed, the four main categories being as follows:

9.2.1 Investigation of the characteristics of raw coal,  
9.2.2 Comprehensive plant and equipment performance tests,

9.2.3 Determination of preparation plant design, and  
9.2.4 Plant control testing.

9.3 The amount of material required, the care to be exercised in performing the washability analysis, and the degree of precision and bias obtained will vary with the purpose for which the analysis is conducted. For this reason, it is paramount that rigid control of the appropriate parameters be exercised throughout the washability procedure.

9.4 As a general guide for a washability analysis of four to six specific gravities, the mass of the gross sample subjected to washability analysis typically is large enough to yield the listed quantity in each size fraction upon sieving as listed in Table 1.

9.4.1 This requirement is usually met by using the gross sample mass resulting from the required mass of the top size fraction; for example, for 100 mm × 600 μm (4 in. × No. 30 USA Standard) size fraction, there should be 910 kg (2000 lb) of 100 mm by 50 mm (4 by 2-in.) material in the sample.

9.4.2 It should also be cautioned that to obtain the necessary mass for a 200 mm × 75 μm (8 in. × No. 200 USA Standard) washability analysis of four to six specific gravities, one cannot simply add the masses found in the right hand column of Table 1. It may be necessary to perform a sieve analysis (see Test Method D 4749) to determine the actual mass that can be expected in each size fraction.

9.5 When taking a bulk sample, it is better to sample

excessively than to have insufficient material. For instance, the masses given in Table 1 may be too small for certain requirements, such as performance testing a preparation plant or where extensive analytical work is required. Because some coals give low yield in middle specific gravity fractions, there may be insufficient material for analytical requirements and the mass of the bulk sample may have to be increased to meet the general guideline of having not less than 20 g nor less than 10 particles in each specific gravity fraction. It may be necessary to increase the mass of the original bulk sample to assure that this guideline is met or, alternatively, specific gravity fractions not meeting this requirement should be added to the appropriate adjacent fraction before weighing and analyzing.

**NOTE 7**—It may be possible, with the smaller size fractions, to complete the washability analysis with less than 20-g recovery per specific gravity fraction, especially if only limited analyses are to be performed upon these fractions. However, due to potential recovery, weighing, and interpretation errors, these data should be cautiously reported and used. In such cases, the actual mass of material recovered from such specific gravity fractions is reported on each analytical report.

9.6 Pilot testing is frequently performed on a representative sample in order to estimate the gross washability characteristics of the bulk materials. The knowledge gained allows for better planning of sampling and washability analysis on a large scale. See Annex A3 for details.

## 10. Preparation of Apparatus

**NOTE 8—Warning:** The organic solutions used in this procedure may be hazardous. See Section 8 and Appendix X1 for precautionary statements.

### 10.1 Coarse-Coal Fractions:

10.1.1 With the wire mesh inserts in place, prepare the large float/sink tanks by filling the tanks three quarters full with the first specific gravity solution that is required for the test.

10.1.2 Establish the desired specific gravity of the solution in accordance with 11.2.

### 10.2 Fine-Coal Fractions:

10.2.1 Prepare the float/sink flasks of 2 to 4-L (0.5 to 1-gal) capacity by filling to within 50 mm (2 in.) of the top of the first specific gravity solution that is required for the test (Note 3).

10.2.2 Establish the desired specific gravity of the solution in accordance with 11.2.

## 11. Calibration and Standardization

11.1 At regular intervals, as dictated by good laboratory practice, calibrate all hydrometers in accordance with Test Method E 126.

11.2 Establish the specific gravity solution in the float/sink vessels using hydrometers calibrated in 11.1.

11.2.1 Carefully adjust the solution to within  $\pm 0.005$  specific gravity unit of the specific gravity desired for the separation by adding lower or higher specific gravity solution as necessary. During the period of testing, check the specific gravity of the solution in the float/sink vessel and adjust as necessary.

11.2.2 Check the solution specific gravity immediately after the testing period. If the reading is beyond  $\pm 0.005$  specific gravity unit of the desired value, change the desired value to

reflect the actual reading, or adjust the solution and repeat the test.

11.2.3 Check the specific gravity by filling a hydrometer cylinder to such a depth as to completely float the hydrometer in the solution. Carefully place the hydrometer into the solution and give a slight spin with the fingers so it settles in the center of the solution without touching the sides of the cylinder. Read the specific gravity to the nearest 0.005 specific gravity unit from the hydrometer scale at the point where it breaks the surface of the solution.

## 12. Procedure A, Coarse Coal

12.1 Carefully place a portion of the coal to be tested into the tank that contains the desired specific gravity solution. All of the coal particles must have ample room to float or sink as their specific gravity dictates. The depth of particles floating should not exceed three to five times the mean diameter of the particles in the sample.

12.2 With occasional stirring of the float and of the sink materials to free all particles, allow these materials sufficient time to either float or sink. Depending upon the size range of the sample, this separation time may be on the order of 5 to 20 min.

12.3 Carefully remove the float material with a wire mesh strainer with mesh size one half the size of the smallest particle, allow to drain, and place this material into a pan. Gently skim off the material, in a manner such that the sink material is not disturbed. Set this float material aside to dry, adequately labeled with its specific gravity float designation.

**NOTE 9**—Monitor and adjust, if necessary, the specific gravity of the solution between each portion of the sample.

12.4 Repeat 12.1-12.3 until the entire sample has been subjected to the desired specific gravity solution.

**NOTE 10—Caution:** The amount of coal floating or sinking at any specific gravity level is a function of the particular coal and the quantity being tested. The amount of sink material in the basket must therefore be monitored so that it does not collect past roughly one-fourth the liquid level within the basket. When the sink material collects near this point, remove in accordance with 12.5 and 12.6 and continue with the remaining coal sample.

12.5 Remove the sink material from the bottom of the float/sink tank by removing the insert that traps the sink material. Allow the insert to remain suspended above the top level of the solution long enough to drain the solution from the material trapped in the insert.

12.6 Remove the sink material from the insert, place this material into a pan, and set the pan and the material aside to dry. Label the material with its specific gravity sink designation.

12.7 Establish the next desired specific gravity solution in accordance with 11.2. Repeat 12.1-12.6 with the appropriate material collected from the preceding specific gravity solution until the gross sample has been incrementally subjected to the series of specific gravities desired for the analysis.

**NOTE 11**—The materials are normally drained between each subsequent specific gravity solution. After the materials are placed into a subsequent specific gravity solution and the float and sink fractions have been removed, the specific gravity of the solution is checked and adjusted, if

necessary, to the proper specific gravity. This is accomplished by following the instructions given in 11.2.3 and if adjustment is necessary, 11.2.1.

12.8 Dry and weigh the float and sink fractions collected from each specific gravity level and process for laboratory analysis in accordance with Method D 2013.

NOTE 12—As a general guide, with good technique, 98 % of the material, on an equivalent moisture basis, should be recovered from each size fraction. Data based on recovery of less than 98 % should be questioned; in all cases, the percent recovery should be reported.

### 13. Procedure B, Fine Coal

13.1 Carefully, and in small increments, add a small portion (up to 200 g) of the coal to be tested into the float/sink flask that contains the desired specific gravity solution. Exercise extreme care to avoid overloading. All of the coal particles must have an opportunity to either float or sink as their specific gravity dictates.

13.2 Thoroughly stir the float and the sink materials in the flask, cover the top of the flask to prevent evaporation of the solution, and allow the two phases to separate into distinct layers. If no separation is apparent after 4 h, the test may be continued with 13.4. If the fine material shows a tendency to travel up the side of the flask, carefully wash the particles back into the solution with a small amount of additional specific gravity solution from a dispenser that contains solution of the same specific gravity as that in the flask.

NOTE 13—Sometimes the fine particles will adhere to or climb the walls of the flask. This can be minimized by spraying the outside of the flask with an anti-static agent.

13.3 If necessary, repeat 13.1 and 13.2 using additional flasks until the entire sample has been placed through the desired specific gravity solution. Alternatively, a single float/sink flask may be used by removing the float material with a wire mesh strainer, adding an additional portion as specified in 13.1, allowing separation to occur as stated in 13.2, and, finally, once the bottom of the float/sink flask is about one quarter filled with sink material, separating the flask and filtering the float and sink materials as instructed in 13.4. (However, this alternative procedure is not good laboratory practice when the increased exposure to the organic liquid results in its absorption, thereby changing the apparent relative specific gravity of the coal, clay, and shale particles or if the intermittent addition and mixing of materials disturbs the float or sink particles, thereby resulting in misplaced materials.) Repeat as needed until the entire sample is treated in this manner (see Note 9).

13.4 Separate the upper and lower portions of the flasks by carefully inserting a stopper attached to a rod into the neck of the flask connections, being careful not to trap any float material on the bottom surface of the stopper.

13.5 With the stopper in place, remove the top portion of the flask from the lower portion, then carefully decant the float material and solution trapped in the upper portion of the flask into a large funnel, pressure filter, or vacuum filter that contains a rapid grade filter paper capable of trapping the float particles.

13.6 With a squeeze dispenser (see 6.4.8) containing solution of the same specific gravity as from 13.5, thoroughly wash any float material adhering to the upper portion of the flask or to the stopper onto the filter paper used to drain the float

material. Filter and dry this float material. Carefully label this material with its proper float designation.

13.7 Carefully decant the solution and sink material in the lower portion of the flask into a large funnel, pressure filter, or vacuum filter that contains a rapid grade of filter paper capable of trapping the sink material.

13.8 With a squeeze dispenser (see 6.4.8) containing solution of the same specific gravity as from 13.7, thoroughly wash any sink material adhering to the lower portion of the flask onto the filter paper used to drain the sink material. Filter and dry this sink material. Carefully label this material with its proper designation.

13.9 Establish the next desired specific gravity solution in accordance with 11.2. Repeat 13.1-13.8 at the next desired specific gravity with the appropriate material collected from the preceding specific gravity until the gross sample has been incrementally subjected to the series of specific gravities desired for the analysis (see Note 11).

13.10 Dry and weigh the float and sink fractions collected from each specific gravity level and process for laboratory analysis in accordance with Method D 2013 (see Note 12).

### 14. Precision and Bias <sup>6</sup>

14.1 Generally, precision and bias statements for samples with a topline larger than 6.3-mm (¼-in.) round cannot be developed for this test method because of the impracticality of obtaining, transporting, and handling representative splits of the materials in the quantities that would be needed to establish the precision and bias statements.

14.2 *Precision*—For samples with a size fraction of 6.3 mm × 1.18 mm (¼-in. round × No. 16 USA Standard), the expected repeatability and reproducibility, based on initial sample weight of 4400 to 6000 g and with specific gravity separations of 1.40 and 1.65 specific gravity, are as follows:

#### 14.2.1 *Recovery*:

Specific Gravity Fraction	Repeatability, g	Reproducibility, g
under 1.40	273	490
1.40 to 1.65	$0.247(W)^A + 59$	$0.296(W)^A + 17$
over 1.65	$0.168(W)^A - 49$	$0.181(W)^A - 16$

<sup>A</sup> *W* = weight of sample recovered at that specific gravity fraction, g.

14.2.1.1 *Example*: Starting with an initial 6000 g sample of 6.3 × 1.18 mm (¼-in. round No. 16 USA Standard) material, 820 g are recovered in the 1.40 to 1.65 specific gravity fraction, the expected repeatability of that fraction is  $(0.247 \cdot 820) + 59$  g or 261.5 g and the expected reproducibility is  $(0.296 \cdot 820) + 17$  or 259.7 g

#### 14.2.2 *Moisture (Applicable Range 0.0 to 7.0 %)*:

Specific Gravity Fraction	Repeatability, %	Reproducibility, %
under 1.40	$0.285(M, \%)^A - 0.03$	$0.559(M, \%)^A + 0.14$
1.40 to 1.65	$0.533(M, \%)^A - 0.39$	$0.626(M, \%)^A + 0.09$
over 1.65	$0.824(M, \%)^A - 0.81$	$0.760(M, \%)^A - 0.12$

<sup>6</sup> Supporting data are available from ASTM Headquarters. Request RR:D05-1010.

<sup>A</sup> *M*, % = moisture content of sample recovered at that specific gravity fraction.

#### 14.2.3 Ash, Dry Basis (Applicable Range 0.0 to 85.0 %):

Specific Gravity Fraction	Repeatability, %	Reproducibility, %
under 1.40	0.38	0.66
1.40 to 1.65	1.07	2.15
over 1.65	2.08	4.29

#### 14.2.4 Sulfur, Dry Basis (Applicable Range 0.0 to 12.0 %):

Specific Gravity Fraction	Repeatability	Reproducibility
under 1.40	0.047( <i>S</i> , %) <sup>A</sup> + 0.028	0.128( <i>S</i> , %) <sup>A</sup>
1.40 to 1.65	0.110( <i>S</i> , %) <sup>A</sup> - 0.072	0.190( <i>S</i> , %) <sup>A</sup> - 0.083

$$\text{over 1.65} \quad 0.125(\text{S, \%})^A - 0.020 \quad 0.199(\text{S, \%})^A + 0.063$$

<sup>A</sup> *S*, % = Sulfur content of sample recovered at that specific gravity fraction.

14.3 *Bias*— Since there is no accepted reference material suitable for determining the bias for the procedure in Test Method D 4371 for measuring washability of coal, bias has not been determined.

## 15. Keywords

15.1 float/sink of coal; relative density; specific gravity; unit density; washability of coal

## ANNEXES

### (Mandatory Information)

#### A1. SELECTION OF PARTICLE SIZE

A1.1 Because of the wide variances in coal handling facilities, in uses of washability data, and in materials that may be encountered in the construction of washability apparatus, it is impractical to specify a particular particle size as being the breakpoint between coarse-coal sizes and fine-coal sizes. For this reason, a range of particle sizes is given. Each application of this washability test method will require the selection of a particular particle size within this range that will serve as the breakpoint between those fractions described and treated as coarse-coal fractions and those fractions described and treated as fine-coal fractions. This particle size should then be used in subsequent washability analyses of the same material sampled from the same location for the same application. In general,

selection of a particle size to be used as the breakpoint between coarse coal and fine coal from outside of this established range would not be advisable because of material handling problems that could then develop, that is, the resulting size fractions would not be amenable to accurate specific gravity separation according to the methods described herein.

NOTE A1.1—It is important to understand that the differentiation between coarse and fine coal, as defined in this test method, is not a differentiation between which coal-size fractions will report to which circuit in a preparation facility, but is simply a differentiation as to how the coal-size fractions will be handled while determining their washability characteristics.

#### A2. ORGANIC LIQUIDS

A2.1 Organic solutions are preferred because the products of the separation are easier to handle and prolonged washing and drying times are unnecessary owing to the relatively low viscosity and the relatively high volatility of the solutions. These organic solutions should be used sparingly and it is recommended that recovery of these solutions be practiced, particularly by drainage after removal of the sample from the separation tank.

A2.2 The volatility of the organic solutions increases the speed of drying of the separated fractions but also results in the loss of the organic solution. This loss is minimized by keeping the tanks covered when not in use. The loss can also be minimized on the fine-coal separations by keeping the float/sink flasks covered while the fine-coal components are separating into their distinct float/sink fractions.

A2.3 The specific gravities of the organic solutions are temperature dependent and will require frequent checking and periodic adjustment. To avoid the need for constantly adjusting the specific gravity, it is advantageous to work in a relatively

constant temperature environment. On occasion, sunlight, hot lights, or air drafts adversely affect the temperature and the specific gravity of the baths.

A2.4 When preparing solutions of these organic liquids for washability analyses, it is important that the specific gravity of the resultant mixture is measured by means of a hydrometer with an accuracy of  $\pm 0.005$  units.

A2.5 It is equally important to monitor the specific gravity of these solutions as they are being used. The specific gravity must be adjusted as necessary between increments. If necessary, increments must be rerun until the proper specific gravity determination is made.

A2.6 In those cases where the end products of the washability analysis will be subjected to further testing that involves the determination of halogen content or where halogen content is an interference in the determination, extreme caution must be exercised to assure that even trace amounts of the organic solutions are removed. Washing with methanol or acetone has

had limited success in removing these solutions used as washability media. All such analytical results from washability samples should be considered suspect unless similar testing, done on samples not having been subjected to washability analysis, supports the results.

A2.7 Consideration must be given to the solvent effect that

some of these organic liquids have upon some coal constituents. This effect is most pronounced in cases where the washability products will be used in determining the suitability of coals for coking. This effect is enhanced if the solutions are not driven off during drying.

### **A3. PILOT TESTING**

A3.1 This procedure is frequently performed on a representative sample in order to estimate the gross washability characteristics of the bulk material. This knowledge enables the operator to plan the actual washability analysis in such a way that unnecessary operations are avoided and the analysis is performed more expeditiously and with less effort.

A3.2 The pilot test or previous knowledge may indicate an advantage in beginning the separation either at the highest or the lowest density solution, or, in some cases, at an intermediate stage in the series of specific gravities to which each size fraction is to be subjected. A sample that gives a high yield at any of these points should be separated at that point so that the bulk of the sample can be removed in one operation. In cases

where there is only a small yield at one or two consecutive specific gravity fractions, it may be better if these fractions are combined before going through a full treatment process.

A3.3 Based upon the above criteria, it is possible to vary the procedure without affecting the outcome of the test, and in many cases, doing so will actually improve its accuracy and reduce the time and labor involved.

A3.4 Pilot testing may be required to establish general washability characteristics of a particular coal in order that subsequent sampling/analysis programs will yield more precise and more reliable results than may be the case without the pilot testing.

## **APPENDIX**

### **(Nonmandatory Information)**

#### **X1. INFORMATION CONCERNING SAFETY PRECAUTIONS REGARDING THE USE OF ORGANIC LIQUIDS**

##### **X1.1 Precautions**

X1.1.1 As the use of organic liquids is subject to safety regulations, and since many of the organic liquids used in these operations may pose serious, acute, and chronic health hazards to individuals working with them, the user of this test method is urged to ensure compliance with relevant regulations.

X1.1.2 The most commonly used organic liquids and their solutions will emit vapors and must be used only in a well-ventilated or hooded area. Where practical, engineering control should be utilized to minimize or eliminate exposure to these chemicals.

X1.1.3 Sampling of personal exposure should take place on individuals using these organic liquids in coal operations to ensure that their exposure without regard to the use of respiratory protective equipment is below the effective threshold limit value (TLV) prescribed for that solvent (10). (Refer to OSHA/MSHA or other pertinent regulations for information on personal exposure.)

X1.1.4 Proper safety equipment must be worn. Relevant regulations regarding fire hazards should be observed. Consult

appropriate references (10, 11,12, 13, 14, 15,16, and 17) for information supplementing the appropriate regulations.

##### **X1.2 Choosing an Alternative Organic Liquid**

X1.2.1 In all cases where a potentially less harmful alternative for any of these organic liquids is available, it should be used if it meets the criteria of inertness, availability, and acceptability. However, all organic liquids, particularly halogenated hydrocarbons, should be considered as suspected carcinogens until proven otherwise. Because a particular organic liquid has not yet been sufficiently tested or has not yet received notoriety is not sufficient justification for its consideration as being harmless.

##### **X1.3 Materials Used in Construction of the Float/Sink Apparatus**

X1.3.1 Aluminum, magnesium, other reactive metals, and their alloys should not be used for the construction of the apparatus as these metals react with some of the halogenated hydrocarbons.

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