



Designation: D 4371 – 91 (Reapproved 1998)

## 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,

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<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.

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 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 ( $\frac{3}{8}$  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 ( $\frac{3}{8}$  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 ( $\frac{3}{8}$  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 ( $\frac{1}{8}$  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.)

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

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.

## 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.

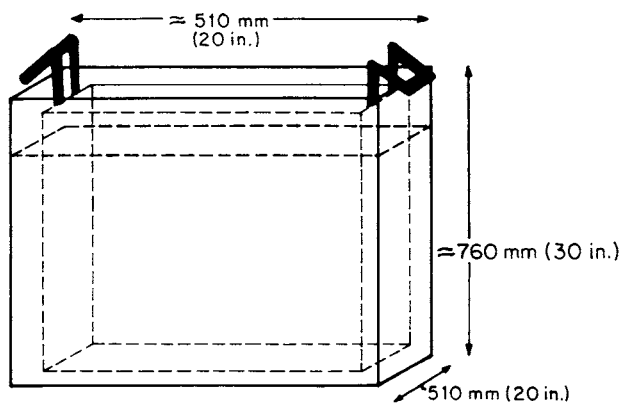
## 7. Reagents and Materials

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.

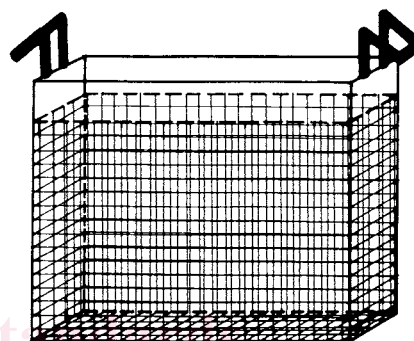
7.1.1 The most suitable range of relative densities is dependent upon the specific purpose of the test and the characteristics

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

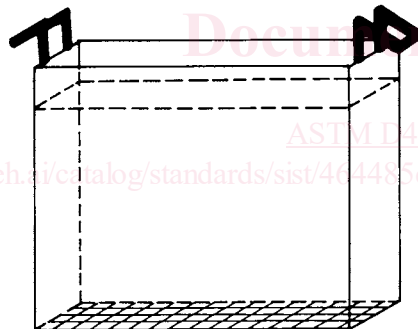
Size Fraction	Mass of Material Needed for Washability Analysis of that Size Fraction, kg (lb)
200 mm $\times$ 100 mm (8 in. $\times$ 4 in. round)	2720 (6000)
100 mm $\times$ 50 mm (4 in. $\times$ 2 in. round)	910 (2000)
50 mm $\times$ 25 mm (2 in. $\times$ 1 in. round)	225 (500)
25 mm $\times$ 12.5 mm (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)



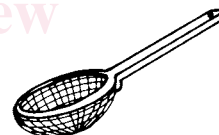
Rectangular Tank with Inner Liner



Rectangular Inner Liner With Wire Mesh Sides and Bottom



Rectangular Inner Liner With Wire Mesh Bottom Only



Wire Mesh Strainer

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

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 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.

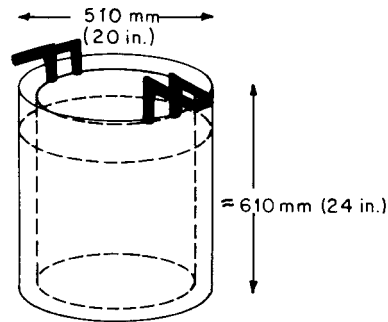
NOTE 6—**Warning:** Some of these organic liquids may be hazardous. See Appendix X1.

## 8. Hazards

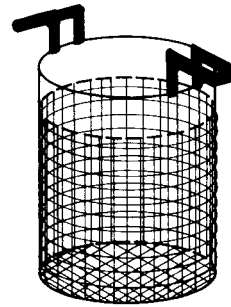
8.1 The most commonly used organic solutions produce vapors and must be used only in a well-ventilated or hooded area. In addition to adequate ventilation, it is also prudent to use appropriate safety measures. (See Appendix X1.)

## 9. Sampling and Test Specimens

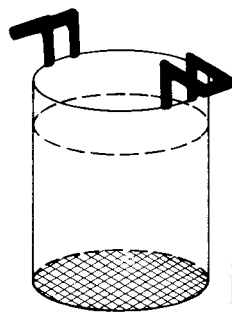
9.1 Obtaining analytical results that truly represent the material being sampled requires planning and diligence in taking the sample and its storage, handling, and preparation (2, 3, 4, 5, 6, 7, 8, 9). The wide varieties of coal handling facilities, the wide differences in physical constraints on the various sampling locations, and the various uses of coal washability data preclude the publication of detailed recommendations for



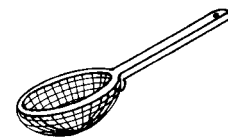
Cylindrical Tank With Inner Liner



Cylindrical Inner Liner With Wire Mesh Sides and Bottom

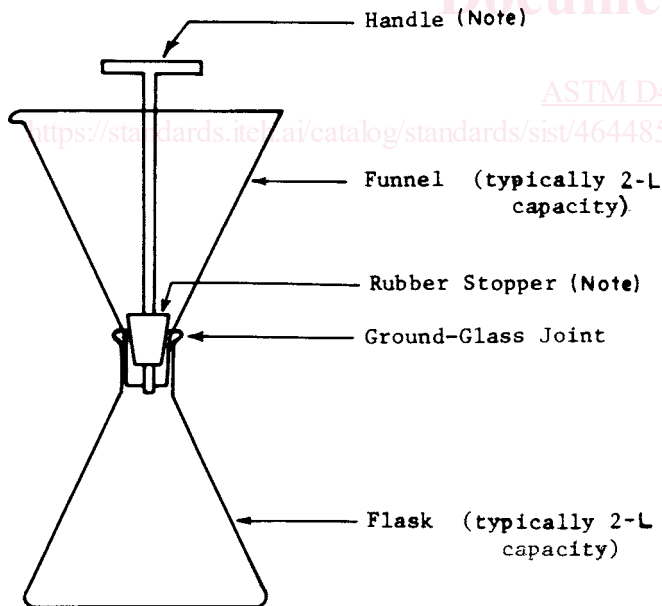


Cylindrical Inner Liner With Wire Mesh Bottom Only



Wire Mesh Strainer

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

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 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 topsize 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.

the sampling of coal for every situation. Sampling and sample preparation, therefore, should be done in accordance with Test

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 ±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 ±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