



Designation: D5114 – 90 (Reapproved 2010)

Standard Test Method for Laboratory Froth Flotation of Coal in a Mechanical Cell¹

This standard is issued under the fixed designation D5114; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Froth flotation of coal, the separation of ash-bearing minerals from combustibles via differences in surface chemistry, has been steadily increasing in use as a means to treat 600- μm (No. 30 U.S.A. Standard Sieve Series) or finer coal. The process is one in which many variables need to be monitored and regulated. Because of this complexity, rigorous laboratory testing is difficult to standardize.

This test method outlines the types of equipment and procedures to apply on a laboratory scale to isolate key process variables and minimize the variations associated with the design and execution of a froth flotation test. The objective of the test method is to develop a means by which repeatable grade/recovery results are ascertained from froth flotation testing of coal without imposing unnecessary limitations on the applicability of the test results in coal preparation practice.

It is recognized that sample preparation, particularly comminution, has a significant impact on froth flotation response. This test method does not attempt to define sample preparation and size reduction practices as part of a froth flotation testing program.

This test method also does not completely cover specific procedures for the investigation of flotation kinetics. Such a test is specialized and is highly dependent upon the end use of the data.

1. Scope

1.1 This test method covers a laboratory procedure for conducting a single froth flotation test on fine coal (that is, nominal top size of 600 μm (No. 30 U.S.A. Standard Sieve Series) or finer) using a defined set of starting point conditions for the operating variables.

1.2 This test method does not completely cover specific procedures for the investigation of flotation kinetics. Such a test is specialized and highly dependent upon the objective of the data.

1.3 Since optimum conditions for flotation are usually not found at the specified starting points, suggestions for development of grade/recovery curves are given in **Appendix X1**. Such a procedure is very case-specific and involves running a series of flotation tests in which some of the operating variables are changed in order to optimize conditions for either yield or grade.

1.4 Laboratory flotation results need not be representative of the flotation response of coal in full-scale situations, but a

consistent baseline can be established against which full-scale performance can be compared.

1.5 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.6 *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.*

1.7 Material Safety Data Sheets (MSDS) for reagents used are to be obtained from suppliers who are to be consulted before work with any chemicals used in this test method.

2. Referenced Documents

2.1 *ASTM Standards:*²

D121 Terminology of Coal and Coke

D2013 Practice for Preparing Coal Samples for Analysis

¹ This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.07 on Physical Characteristics of Coal.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- D2015 Test Method for Gross Calorific Value of Coal and Coke by the Adiabatic Bomb Calorimeter (Withdrawn 2000)³
- D2234/D2234M Practice for Collection of a Gross Sample of Coal
- D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke
- D3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal
- D3177 Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke (Withdrawn 2012)³
- D4239 Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High-Temperature Tube Furnace Combustion
- D4749 Test Method for Performing the Sieve Analysis of Coal and Designating Coal Size

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology D121.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *collector*—a reagent used in froth flotation to promote contact and adhesion between particles and air bubbles.

3.2.2 *combustibles*—the value obtained by subtracting the dry weight (in percent) of the ash (as determined in Test Method D3174) from 100 % representing the original weight of the analyzed sample.

3.2.3 *concentrate*—the froth product recovered in coal froth flotation.

3.2.4 *conditioning agents*—all chemicals that enhance the performance of the collectors or frothers. Conditioning agents change the characteristics of the surface of the minerals or the environment. There are many subgroups according to function: activators, depressants, emulsifiers, dispersants, flocculants, chelating reagents, froth depressants, pH modifiers, and so forth.

3.2.5 *flotation cell*—the vessel or compartment in which the flotation test is performed.

3.2.6 *froth*—a collection of bubbles and particles on the surface of a pulp in a froth flotation cell.

3.2.7 *froth flotation*—a process for cleaning fine coal in which hydrophobic particles, generally coal, attach to air bubbles in a water medium and rise to the surface to form a froth. The hydrophilic particles, generally the ash-forming matter, remain in the water phase.

3.2.8 *frother*—a reagent used in froth flotation to control the size and stability of the air bubbles, principally by reducing the surface tension of water.

3.2.9 *grade/recovery*—the relationship between quality and quantity of the clean coal product. The quality can be defined in terms of ash, sulfur, or Btu content. The quantity can be designated as yield or heating value recovery (Btu or combustibles).

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

3.2.10 *mechanical cell*—a type of flotation cell that employs mechanical agitation of a pulp by means of an immersed impeller (rotor) and stator stirring mechanism. Aeration to the cell can be from an external pressurized air source or self-induced air.

3.2.11 *natural pH*—the measured pH of the pulp prior to the addition of collector, frother, or any conditioning agents.

3.2.12 *pulp*—a fluid mixture of solids and water, also known as slurry.

3.2.13 *recovery*—the percent of the valuable component (that is, Btu or combustible) from the feed that reports to the froth concentrate product.

3.2.14 *solids concentration*—the ratio, expressed as a percent, of the weight (mass) of solids to the sum of the weight of solids plus water.

3.2.15 *tailings*—the underflow product from coal froth flotation.

3.2.16 *yield*—the weight percent of the feed that reports to the concentrate.

4. Significance and Use

4.1 This test method uses specific starting point conditions for the froth flotation response to accomplish the following:

4.1.1 Assess responses of one or more coals or blends of coal, and

4.1.2 Evaluate and determine froth flotation circuit performance.

5. Apparatus

5.1 *Laboratory Flotation Machine*, with a minimum volume of 2 L and a maximum volume of 6 L. Fig. 1 schematically

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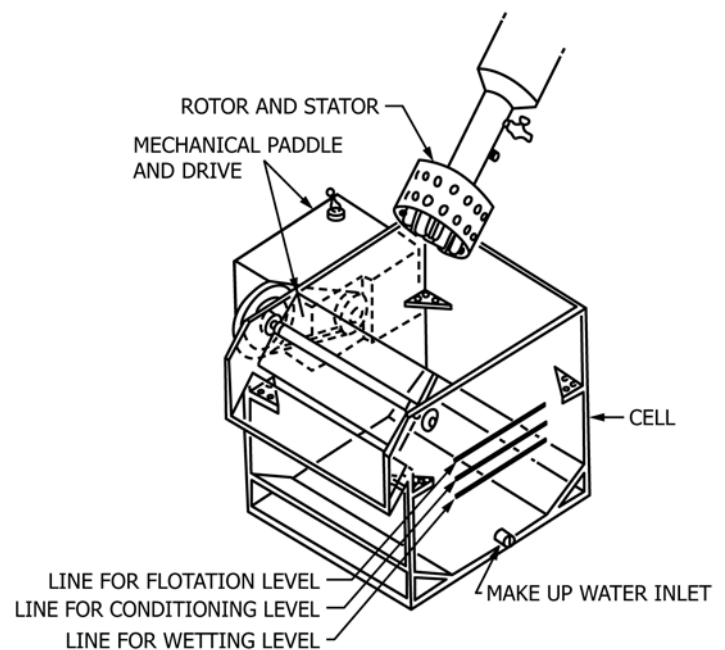


FIG. 1 5.5-L Mechanical Paddle Laboratory Froth Flotation Cell

depicts a batch mechanical flotation cell⁴ which can be used in conjunction with this test method. The major criterion is that the unit must be able to provide for constant mechanical removal of froth from the cell. In addition, the laboratory unit must have some means of automatic liquid level control.

5.1.1 An example of a mechanical paddle laboratory froth flotation apparatus is shown in Fig. 1. The froth paddles are rotated at approximately 30 r/min, thus avoiding variation caused by manual removal of froth. The froth paddle shall not rotate below the pulp surface and not more than 6 mm (¼ in.) above the pulp level. The distance between the overflow lip and the edge of the froth paddle shall be at least 3 mm (⅛ in.) but not more than 6 mm (¼ in.).

5.1.2 The pulp in the cell is maintained at a constant level by a small tank with an overflow at precisely the desired level to be maintained in the flotation cell.

NOTE 1—Another suitable slurry level control system consists of a resistance type level probe, a resistance sensor relay, a solenoid valve, and associated connecting wires.⁵ The level probe is mounted inside the cell and is connected to the resistance relay which operates the solenoid valve. When the slurry level drops below the tip of the probe, the relay energizes the solenoid valve. Then, makeup water flows into the cell. When the level rises up to the probe, the solenoid valve is de-energized, which stops the makeup water flow.

5.2 *pH Meter*, sensitive to 0.1 units.

5.3 *Timing Device* that displays cumulative minutes and seconds.

5.4 *Air Flow Meter*.

5.5 *Microsyringes or Pipets*.

5.6 *Balances*, with a readability of at least 0.5 % of the total weight.

5.7 *Vacuum or Pressure Filter*, or a filter funnel for gravity filtration.

5.8 *Drying Oven* with forced air, capable of maintaining a maximum temperature of 40°C (104°F) and meeting the requirements of Method D2013.

5.9 *Rinse Bottle*.

6. Sample Preparation

6.1 The sample history, moisture content, alteration of the inherent moisture, or alteration of the surface properties have considerable effect on the flotation characteristics of the coal. It is important that all samples used in flotation testing are stored and handled so as to minimize alteration of the surface properties. The origin and history of the sample should be recorded. It is imperative that all samples be prepared in a similar manner. Since the generation of grade/recovery curves will involve several individual tests, sample subdivision and preparation must be carefully performed to ensure that each subsample is representative of the original whole sample.

⁴ A suitable cell, available from WEMCO, 1796 Tribute Rd., Sacramento, CA 95815, or equivalent can be used.

⁵ A suitable slurry level control system, available from C&R Technology, Inc., P.O. Box 114, Fall Branch, TN 37656, or equivalent can be used.

7. Flotation Conditions

7.1 The conditions under which a test program is conducted will be systematically varied to generate grade/recovery curves (Appendix X1). Table 1 outlines recommended starting point conditions for a single laboratory-scale test. These conditions are for laboratory testing parameters and are not designed to simulate in-plant operating conditions that can be highly variable, such as water temperature and chemistry.

7.2 *Slurry Temperature*—The operating temperature shall be $22 \pm 5^\circ\text{C}$ ($72 \pm 9^\circ\text{F}$).

7.3 *Water*—Plant, tap, or distilled water may be used, whichever is consistent with the object of the test. The source of water must be recorded.

7.4 *Solids Content*—The solids content corresponds with that of the industrial preparation plant slurry, if the object of the test is to simulate plant conditions. Otherwise, an 8 % solids concentration shall be used.

7.5 *Pulp Level*—Maintain between 12.7 and 15.9 mm (0.50 and 0.62 in.) below the lip of the cell as measured with the air on and stirrer operating.

7.6 *Wetting of Coal*—Before the addition of reagents and subsequent flotation, it is important to ensure that the proper air bubble attachment can take place at the coal-water interface. Wetting is accomplished in the cell by running the impeller at the r/min specified for the flotation step with the air off. Perform this step for 5 to 10 min before reagent addition. If the sample is in slurry form this wetting step is not necessary.

7.7 *Reagent Addition*—Collector, frother, conditioning agent, or any combination thereof shall be governed by the requirements of the test. Add reagents to the coal slurry and condition to ensure proper distribution of reagents. Conduct the conditioning step at the same impeller speed as the flotation step with the air flow off.

7.7.1 Add the reagents using either a calibrated microsyringe or a pipet.

7.8 *Air Flow*—Rate shall be measured and recorded.

7.9 *Impeller Speed*—The starting speed shall be 1200 r/min.

NOTE 2— Impeller speed is an important variable and should be investigated during optimization, depending on the object of the test.

8. Procedure

8.1 Calculate the total mass of coal required for the number of flotation tests based on the measured cell volume and the test solids content.

8.2 Divide the total mass into representative portions by riffing, in accordance with Method D2013. A few small increments, totalling no more than 15 % of the total mass, may be either taken from the subsample or added to the subsample in order to obtain the exact weight.

8.3 Determine the particle size distribution of one of the portions from 8.2 in accordance with Test Method D4749.

8.4 Rinse the cell thoroughly with water. Add from one half to two thirds of the total required water to the cell. Confirm that the air is turned off. Turn the impeller on and adjust to the