



Designation: D1857/D1857M – 17a

## Standard Test Method for Fusibility of Coal and Coke Ash<sup>1</sup>

This standard is issued under the fixed designation D1857/D1857M; 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.

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

### 1. Scope

1.1 This test method covers the observation of the temperatures at which triangular pyramids (cones) prepared from coal and coke ash attain and pass through certain defined stages of fusing and flow when heated at a specified rate in controlled, mildly reducing, and where desired, oxidizing atmospheres.

1.2 The test method is empirical, and strict observance of the requirements and conditions is necessary to obtain reproducible temperatures and enable different laboratories to obtain concordant results.

1.3 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.

1.3.1 All percentages are percent mass fractions unless otherwise noted.

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.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of D05.21 on Methods of Analysis.

Current edition approved Aug. 1, 2017. Published August 2017. Originally approved 1961. Last previous edition approved in 2017 as D1857 – 17. DOI: 10.1520/D1857-17a.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

D2013 Practice for Preparing Coal Samples for Analysis

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

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

D3682 Test Method for Major and Minor Elements in Combustion Residues from Coal Utilization Processes

D4326 Test Method for Major and Minor Elements in Coal and Coke Ash By X-Ray Fluorescence

D6349 Test Method for Determination of Major and Minor Elements in Coal, Coke, and Solid Residues from Combustion of Coal and Coke by Inductively Coupled Plasma—Atomic Emission Spectrometry

D7448 Practice for Establishing the Competence of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke

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

### 3. Terminology

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

3.1.1 The critical temperature points to be observed are as follows:

3.1.2 *fluid temperature, FT*—the temperature at which the fused mass has spread out in a nearly flat layer with a maximum height of 1.6 mm [ $1/16$  in.] as shown by the fifth cone, FT, in Fig. 1.

3.1.3 *hemispherical temperature, HT*—the temperature at which the cone has fused down to a hemispherical lump at which point the height is one half the width of the base as shown by the fourth cone, HT, in Fig. 1.

3.1.4 *initial deformation temperature, IT*—the temperature at which the first rounding of the apex of the cone occurs. Shrinkage or warping of the cone ignored if the tip remains sharp. In Fig. 1, the first cone shown is an unheated one; the second cone IT is a typical cone at the initial deformation stage.

3.1.5 *softening temperature, ST*—the temperature at which the cone has fused down to a spherical lump in which the height is equal to the width at the base as shown by the third cone, ST, in Fig. 1.

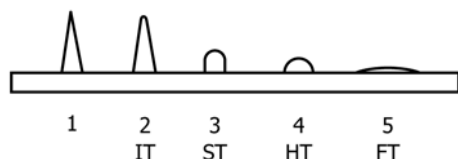


FIG. 1 Critical Temperature Points

#### 4. Summary of Test Method

4.1 Coal or coke ash, prepared by prescribed methods, is mixed with a small amount of a dextrin solution to form a plastic mass. A cone mold is used to form triangular pyramids with the plastic mass, which are dried and mounted on a ceramic platform. The cones and platform are rapidly heated in an electric furnace to 400 °C (750 °F). The cones and platform are then heated at a rate of 8 °C ± 3 °C [15 °F ± 5 °F]/min in a reducing (CO/CO<sub>2</sub>) atmosphere, or an oxidizing (air) atmosphere. The ash cones are carefully observed as they pass through certain defined stages of melting and flow.

#### 5. Significance and Use

5.1 The design of most coal combustion and coal conversion equipment anticipates that the ash either remain solid or assume some degree of fluidity, depending on the particular design. Ash fusibility temperatures help predict whether the ash will perform properly in the process for which the coal was chosen.

5.2 Ash fusibility temperature values are used in various equations to predict the slagging tendency of ashes.

#### 6. Apparatus

6.1 *Furnace*—An electric furnace conforming to the following requirements may be used:

6.1.1 Capable of maintaining a uniform temperature zone in which to heat the ash cones.

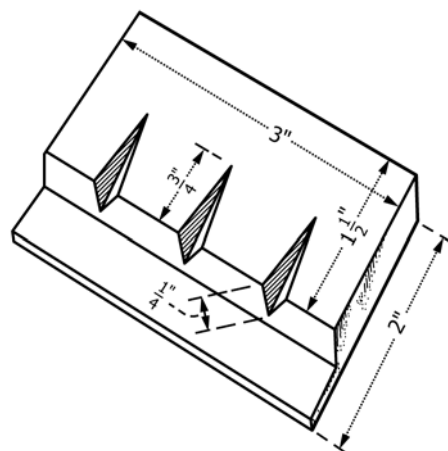
6.1.2 Capable of maintaining the desired atmosphere surrounding the cones during heating. The composition of the atmosphere, reducing or oxidizing, shall be maintained within the limits specified in Section 7. The desired atmosphere is to be obtained by means of gases introduced into the heating chamber. The furnace should be gas impervious.

6.1.3 Capable of regulation so that the rate of temperature rise shall be 8 °C ± 3 °C [15 °F ± 5 °F]/min.

6.1.4 Providing means of observing the ash cones during the heating. Observation on the same horizontal plane as the cone-support surface shall be possible.

6.2 *Cone Mold*—A commercially available cone mold, typically brass or made from a corrosion-resistant material with a low coefficient of friction, as shown in Fig. 2. The cone shall be 19 mm [¾ in.] in height and 6.4 mm [¼ in.] in width at each side of the base, which is an equilateral triangle. A steel spatula with a pointed tip, ground off to fit the cone depression in the mold, is suitable for removal of the ash cone.

6.3 *Refractory Tray Mold*—A mold with flat top and bottom surfaces to provide a refractory tray of suitable thickness to minimize warping. A sidemold not over 6.4 mm [¼ in.] high of any convenient shape, placed on an iron plate so that the top



| Inch-pound<br>Units,<br>in. | SI Units,<br>mm |
|-----------------------------|-----------------|
| 1/4                         | 6.4             |
| 3/4                         | 19.1            |
| 1 1/2                       | 38.1            |
| 2                           | 50.2            |
| 3                           | 76.2            |

FIG. 2 Brass Cone Mold

surface of the refractory mix can be struck off flat and parallel to the tray by means of a straight edge, is satisfactory. Legs not over 3 mm [⅛ in.] long may be provided on the corners of the cone tray by suitable holes bored in the iron base plate of the mold.

6.4 *Temperature Measuring Instrument*—A type R or S thermocouple of platinum and platinum-rhodium, protected from the furnace gases by a glazed porcelain sheath, shall be used with a display readable to better than 5.5 °C [10 °F]. The hot junction of the thermocouple shall touch the end of the sheath and shall be located in the center of the furnace and immediately to the rear of the cones. The thermocouple protective sheath shall be checked for cracks when there is difficulty in achieving the requirements outlined in Section 10.

#### 7. Reagents and Materials

7.1 *Ash-Cone Refractory Trays*—The ash cones shall be mounted on refractory trays. These trays are commercially available, or can be prepared using a mixture of equal parts by weight of kaolin and alumina. Materials conforming to the following requirements are to be used in preparing the refractory trays:

7.2 *Kaolin*—NF-grade powder passing a 75 µm (U.S.A. Standard No. 200) sieve.

7.3 *Aluminum Oxide*—Reagent grade ignited powder passing a 150 µm (U.S.A. Standard No. 100) sieve.

7.4 *Dextrin Solution*—clear, filtered (if necessary) 10 % solution of dextrin containing 0.1 % salicylic acid as a preservative.

7.5 *Mold Release Agent*—petroleum jelly, thinned with kerosene (if necessary), or a non-silicon-based mold release agent.

7.6 *Gold Wire*—0.51 mm diameter (twenty-four gage) or larger round wire of 99.98 % purity, but drawn from metal of 99.99 % purity, and having a melting point of 1064 °C [1948 °F].<sup>3</sup>

7.7 *Nickel Wire*—0.51 mm diameter (twenty-four gage) or larger round wire of CP nickel, 99.98 % pure, fully annealed, and having a melting point of 1455 °C [2651 °F].<sup>3</sup>

## 8. Test Atmospheres

8.1 *Reducing Atmosphere Test*—A regulated flow of gas of the nominal composition, volume fraction 60 % carbon monoxide and volume fraction 40 %  $\pm$  5 % carbon dioxide, shall be maintained in the heating chamber throughout the test (Note 1) in the furnace. The gas stream shall be regulated by any convenient means to provide a measured flow of 1.3 to 1.5 furnace volumes per minute.

8.2 *Cylinders of CO/CO<sub>2</sub> Gas*—To assure that the CO/CO<sub>2</sub> gas remains mixed, maintain the temperature of the cylinder above the critical temperature at which CO<sub>2</sub> can liquefy and separate.

8.3 *Oxidizing Atmosphere Test*—A regulated stream of air shall be maintained throughout the test in the furnace. The gas stream shall be regulated by any convenient means to provide a measured flow of 1.3 to 1.5 furnace volumes per minute.

## 9. Sample and Test Specimen Preparation

9.1 *Coal and Coke*—Prepare the analysis sample in accordance with Practice D2013 for coal or Practice D346 for coke by pulverizing the material to pass a 250  $\mu$ m (No. 60) U.S.A. standard sieve.

9.2 *Laboratory Ashing of Coal or Coke Analysis Sample*—Prepare the ash from a thoroughly mixed analysis sample of coal or coke (see 9.1). To facilitate the ashing process, spread the coal or coke in a layer about 6 mm ( $\frac{1}{4}$  in.) in depth in a porcelain, quartz, fused silica, or fireclay roasting dish. Place the dish in a muffle furnace at ambient temperature and heat gradually so that the temperature reaches 500 °C  $\pm$  10 °C at the end of 1 h. For coals, continue heating the sample until the temperature rises from 500 °C  $\pm$  10 °C to 750 °C  $\pm$  15 °C in 1 h. For cokes, continue heating the sample until the temperature rises from 500 °C  $\pm$  10 °C to 950 °C  $\pm$  10 °C in 1 h. Continue to heat at the final temperature (750 °C or 950 °C) until the test specimen reaches a constant mass or for an additional 2 h (Note 1). Allow the dish to cool, transfer to an agate mortar (Note 2), and grind to pass a 75  $\mu$ m (No. 200) U.S.A. standard sieve. Reignite the ash at 750 °C or 950 °C for 1½ h to ensure complete and uniform oxidation of the ash.

9.2.1 The two-stage ashing procedure allows pyritic sulfur to be oxidized and sulfur oxides expelled before most metal carbonates are decomposed. An ample supply of air in the muffle furnace must be assured at all times to ensure complete oxidation of the pyritic sulfur and, more importantly, to efficiently remove the SO<sub>2</sub> formed. Because of the possibility of SO<sub>2</sub> product being absorbed by alkali and alkaline earth metal oxides (especially CaO), it is advisable to not ash high

pyritic sulfur coals with coals that have high alkali or alkaline earth metal contents, or both.

NOTE 1—The D3174 and D7582 ashing procedures used in the determination of ash from coal and/or coke samples and the D3682, D4326, and D6349 ashing procedures used in preparing ash from coal or coke samples, or both, for major and minor element analysis are acceptable procedures for preparing ash for the fusibility test.

NOTE 2—A mechanical agate mortar grinder will save time where many determinations are made.

9.3 *Preparation of Cones*—Thoroughly mix the ignited ash in a mechanical mixer or on a sheet of glazed paper or oil cloth by raising first one corner to roll the ash over and then raising each of the other corners in rotation in the same manner until each corner has been raised five times or more.

9.3.1 Take sufficient ash for the number of cones desired from various parts of the bulk ash. Moisten the ash with a few drops of a dextrin solution and work it into a stiff plastic mass with a spatula. Press the plastic material firmly with a spatula into the cone mold to form the triangular pyramids. Strike off the exposed surfaces of the material smooth and remove the cones from the mold by applying slight pressure at the base with a suitably pointed spatula. Previous coating of the mold with a very thin layer of petroleum jelly, thinned with kerosene (if necessary), or a very thin coating of a non-silicon-based mold release agent, aids in preventing adherence of the cones to the mold and in providing the sharp point and edges desired in the cone. With certain coal ashes, cones with sharp points and edges can be obtained using distilled water in place of the dextrin solution and without the use of petroleum jelly.

9.3.2 Place the cones in a suitable location to dry sufficiently to permit handling without deformation. Mount the dried cones vertically on a refractory tray.

9.3.2.1 Follow the manufacturer's recommendations for mounting the dry cones on commercially available support trays.

9.3.2.2 To prepare fresh support trays, moisten a portion of the well-mixed kaolin-alumina mixture with the minimum amount of water to make a workable, but stiff, plastic mass, and firmly press it into the support mold (Section 7.4). Strike off the surface of the mass flat and smooth with a steel spatula, moistening with one or two drops of water if necessary to obtain a smooth surface. A number of cones may be mounted on one base. Make shallow triangular depressions, not over 0.8 mm [ $\frac{1}{32}$  in.] in depth, with a triangular file ground to the correct size to produce a depression to fit the base of the cone, and locate the cones sufficiently distant from adjacent cones so that no merging of the fusing material of the cones shall occur during the test. Mount the cones vertically in the depressions while the base is still wet without the use of ash or refractory as a mounting aid (Note 3 and Note 4).

NOTE 3—The intent of the triangular depression is to enable the cones to be mounted in a sufficiently stable manner to permit handling of the prepared support with cones.

NOTE 4—Gold wires can be mounted on each cone support beside the ash cones, and the gold melting point observed concurrent with the ash cones in both oxidizing and reducing atmospheres.

9.4 Before running the test in a reducing atmosphere, dry the mounted cones and ignite (750 °C) for 1 h to remove all carbonaceous material. If no organic material has been used in

<sup>3</sup> These temperatures are consistent with the ITS-90 temperature scale.

the preparation of the mounted cones, this ignition step that follows the drying step may be omitted. The drying step must be retained.

## 10. Furnace Permanent Check

### 10.1 *Furnace Performance Check by Internal Reference Material*

10.1.1 Refer to Test Method **D7448** A1.9.7.2 for the definition of and requirements for an internal reference material.

10.1.2 Monitor the critical temperature points of the internal reference material for each atmosphere according to Test Method **D7448** Section A1.12. Ensure that the checks address all test positions within the furnace. At least one internal reference material shall be analyzed along with each batch of 20 or fewer samples (that is, internal reference materials shall comprise a minimum of 5 % of each set of samples).

10.1.2.1 If out of control conditions are observed in the critical temperatures of the internal reference material, in either atmosphere, proceed to check the furnace temperature by subsection **10.2**.

### 10.2 *Furnace Temperature Check by Gold Melting Point Observation and Reducing Atmosphere Check by Nickel Melting Point Observation*

10.2.1 Insert a support with mounted pieces of gold and nickel wire (12.7 mm [ $\frac{1}{2}$  in.] lengths) into the test furnace. Locate it at the position used for tests of ash cones.

10.2.2 Establish the reducing gas atmosphere and heat the furnace chamber in accordance with **11.1**.

10.2.3 Observe the temperatures shown on the instrument display when the pieces of wire melt.

10.2.4 The observed melting points shall be within  $\pm 5.5$  °C [ $10$  °F] of the following:

|        |                   |
|--------|-------------------|
| Gold   | 1064 °C [1948 °F] |
| Nickel | 1455 °C [2651 °F] |

10.2.5 If the indicated melting point for gold wire falls outside of the desired range, recalibrate the temperature measuring instrument, adjust the depth of sample insertion, or both recalibrate and adjust, so that the average temperature from several observations of the gold melting point is within the specified range.

10.2.6 If the indicated melting point for nickel wire falls outside the desired range, after applying the corrections as described in **10.2.5**, the constancy of the indicated temperatures and the subsequent appearance of the specimen should be examined closely. Erratic readings or failures to obtain melting at 1455 °C [2651 °F] can be due to nickel oxidation caused by an insufficient reducing atmosphere. If a consistent error of more than 14 °C [25 °F] is found, the furnace atmosphere and the temperature measurement equipment may be at fault. Remedial action is required.

## 11. Procedure

### 11.1 *Reducing Atmosphere Test:*

11.1.1 Place the mounted test cones in the furnace at a temperature of not over 400 °C [750 °F] for the furnace to provide sufficient time to purge the air from the uniform temperature zone and establish the desired atmosphere.

11.1.2 If the furnace temperature is below the temperature specified in **11.1.1**, raise it rapidly to the specified temperature before introducing a reducing gas atmosphere. Then control the rate of heating to give a rate of temperature increase of  $8$  °C  $\pm$   $3$  °C [ $15$  °F  $\pm$   $5$  °F]/min. Maintain this rate throughout the test.

### 11.1.3 *Oxidizing Atmosphere Test:*

11.1.3.1 Place the mounted test cones in the furnace at a temperature of not over 400 °C [750 °F] for the furnace. If the furnace temperature is below the temperature specified, raise it rapidly to the specified temperature, then control the rate of heating to give a rate of temperature increase of  $8$  °C  $\pm$   $3$  °C [ $15$  °F  $\pm$   $5$  °F]/min. Maintain this rate throughout the test.

11.1.3.2 Establish the oxidizing atmosphere surrounding the cones, as specified in **8.3** at the temperature specified in **11.1.3.1**. Maintain this atmosphere throughout the test.

## 12. Precision

12.1 The precision data for the determination of the fusion temperatures of coal and coke ash are shown **Table 1**.

**TABLE 1 Repeatability and Reproducibility for Fusion Temperatures of Coal and Coke Ash**

|                                     | °F  | °C |
|-------------------------------------|-----|----|
| Repeatability Limit (r)             | 50  | 30 |
| Reproducibility Limit (R)           |     |    |
| For reducing atmosphere:            |     |    |
| Initial deformation temperature, IT | 125 | 70 |
| Softening temperature, ST           | 100 | 55 |
| Hemispherical temperature, HT       | 100 | 55 |
| Fluid temperature, FT               | 150 | 85 |
| For oxidizing atmosphere:           |     |    |
| Initial deformation temperature, IT | 100 | 55 |
| Softening temperature, ST           | 100 | 55 |
| Hemispherical temperature, HT       | 100 | 55 |
| Fluid temperature, FT               | 100 | 55 |

12.1.1 *Repeatability (r)* the value below which the absolute difference between two test results of separate and consecutive test determinations, carried out on the same sample in the same laboratory by the same operator using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %.

12.1.2 *Reproducibility (R)* the value below which the absolute difference between two test results calculated to a dry basis (Practice **D3180**) carried out in different laboratories using samples taken at random from a single quantity of material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

## 13. Keywords

13.1 ash; coal; coke





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