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Standard Test Method for Fusibility of Coal and Coke Ash[1]

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 (ε) 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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
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, denoting the atmosphere used:~~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.

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[1] 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.

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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.

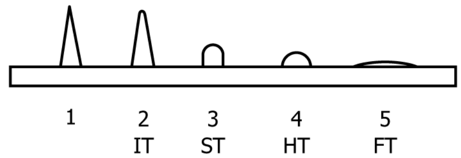


FIG. 1 Critical Temperature Points

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.

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_2) 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 ~~Design~~ 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 ~~and Materials~~

6.1 *Furnace*—~~Any gas-fired or~~ 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. ~~This zone shall be such that the difference in the melting point of 12.7-mm [½-in.] pieces of pure gold wire when mounted in place of the ash cones on the cone support shall be not greater than 11°C [20°F] in a reducing atmosphere test run.~~

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 ~~6~~7. The desired atmosphere ~~in the gas-fired furnace surrounding the cones shall be obtained by regulation of the ratio of gas to air in the combustion mixture. The desired atmosphere in the electric furnace shall~~ is to be obtained by means of gases introduced into the heating chamber. The ~~muffle shall be gas impervious, free from cracks, and the closure plug tight-fitting. The gas supply tube shall be sealed to the back wall of the preheating chamber and shall not extend to the front of the preheating chamber against the perforated baffle.~~ furnace should be gas impervious.

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

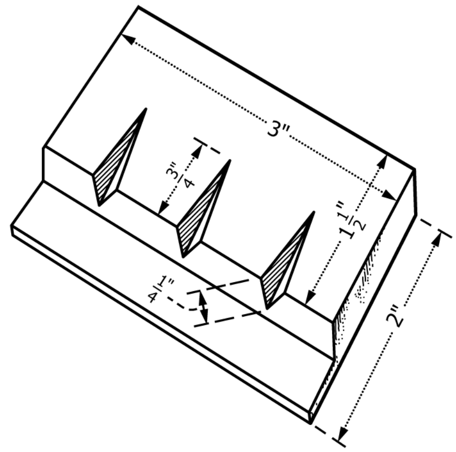
6.2 *Cone Mold*—A commercially available cone ~~mold~~ mold, typically brass or made from a corrosion-resistant material with a low coefficient of friction, as shown in Fig. 2. The cone shall be 19 mm [¾ in.] in height and 6.4 mm [¼ in.] in width at each side of the base, which is an equilateral triangle. 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.

~~5.3 *Optical Pyrometer or Thermocouple,* for temperature measurements, conforming to the following requirements:~~

~~5.3.1 *Optical Pyrometer*—An optical pyrometer of the disappearing filament type shall be used. The instrument shall have been calibrated to be accurate within 11°C [20°F] up to 1400°C [2550°F] and within 16°C [30°F] from 1400 to 1600°C [2550 to 2900°F] (Note 1). The pyrometer filament shall be sighted on the cones until the softening point temperature *C* (Fig. 1) has been passed, and then sighted on the cone support. The pyrometer shall have readable graduations not larger than 5.5°C [10°F].~~

~~NOTE 1—The pyrometer equipment shall be standardized periodically by a suitably equipped standardizing laboratory such as that of the National Bureau of Standards, or checked periodically against equipment certified by the Bureau of Standards.~~

~~5.3.2 *Thermocouple*—A thermocouple of platinum and platinum-rhodium, protected from the furnace gases by a glazed porcelain sheath, shall be used with a high-resistance millivoltmeter or potentiometer accurate and readable to within 5.5°C [10°F]. The sheath shall be sealed to the furnace wall by alundum cement. The hot junction of the thermocouple shall touch the end of the sheath and shall be located in the center of the muffle and immediately to the rear of the cones. The thermocouple protective sheath shall be checked periodically for cracks. The thermocouple and its meter shall meet the requirements of Section 9. The potentiometer or millivoltmeter shall be so located or adequately shielded as to prevent radiant and convection heating of the cold junction. The room temperature compensator shall be adjusted to the existing temperature.~~



Inch-pound Units, in.	SI Units, mm
¼	6.4
¾	19.1
1½	38.1
2	50.2
3	76.2

FIG. 2 Brass Cone Mold

~~5.4 *Ash-Cone Refractory Support*—The ash cones shall be mounted on a refractory base composed of a mixture of equal parts by weight of kaolin and alumina conforming to the following requirements:~~

~~5.4.1 *Kaolin*—NF-grade powder passing a 75-μm (No. 200) sieve.~~

~~5.4.2 *Aluminum Oxide*—Reagent grade ignited powder passing a 150-μm (No. 100) sieve.~~

6.3 *Refractory ~~Support~~Tray Mold*—A mold with flat top and bottom surfaces to provide a refractory ~~support~~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 surface of the refractory mix can be struck off flat and parallel to the ~~base~~tray by means of a ~~straightedge,~~ straight edge, is satisfactory. ~~For electric furnace use, legs not over~~ Legs not over 3 mm [⅛ ~~in. [3 mm]~~in.] long may be provided on the corners of the cone ~~support~~tray by suitable holes bored in the iron base plate of the mold.

6.4 *~~Gold Wire~~Readout Device*—~~—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 1063°C [1945°F].~~A thermocouple of platinum and platinum-rhodium, protected from the furnace gases by a glazed porcelain sheath, shall be used with an appropriate readout device accurate and readable to within 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 periodically for cracks. The thermocouple and its meter shall meet the requirements of Section 10.

~~5.7 *Nickel Wire*[4]—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 1452°C [2645°F].~~

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 1063 °C [1945 °F].

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 1452 °C [2645 °F].

8. Test AtmosphereAtmospheres

6.1 *Gas-Fired Furnace:*

6.1.1 *Reducing Atmosphere Test*—A mildly reducing atmosphere surrounding the cones shall be maintained during the test in the gas-fired furnace. Hydrogen, hydrocarbons, and carbon monoxide shall be considered as reducing gases; oxygen, carbon dioxide, and water vapor shall be considered as oxidizing gases. Nitrogen is inert. The ratio by volume of reducing gases to oxidizing gases in the atmosphere shall be between the limits of 20 to 80 and 80 to 20,[5] that is, on a nitrogen-free basis, the total amount of reducing gases present shall be between the limits of 20 and 80 volume %. A flame 152 to 203 mm [6 to 8 in.] in height and tinged with yellow above the furnace outlet has been found to provide an atmosphere within the specified limits.

6.1.2 *Oxidizing Atmosphere Test*—An atmosphere containing a minimum amount of reducing gases shall be maintained surrounding the cones during the test in the gas-fired furnace. On a nitrogen-free basis, the volume of reducing gases present in the atmosphere shall not exceed volume 10 %. Combustion with the maximum possible quantity of air with preservation of the specified rate of temperature increase has been found to provide an atmosphere within the specified limits. A completely blue flame, not over 50 mm [2 in.] in height above the outlet at the beginning of the test, provides the desired atmosphere; and, by regulation of the combustion gas-air ratio, the specified atmosphere and temperature rise can be maintained.

8.1 *Electric Furnace: Reducing Atmosphere Test*—

6.2.1 *Reducing Atmosphere Test*—A regulated flow of gas of the nominal composition, 60 % carbon monoxide and 40 ± 5 volume % carbon dioxide,[6] shall be maintained in the heating chamber throughout the test (Note 2) in the electric 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. A regulated flow of gas of the nominal composition, volume fraction 60 % carbon monoxide and volume fraction 40 ± 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.

6.2.2 *Oxidizing Atmosphere Test*—A regulated stream of air shall be maintained throughout the test in the electric 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.

NOTE 2—Before using new cylinders of CO/CO_2 reducing gas, the contents should be mixed according to the gas manufacturer's recommendations. To assure that the gas remains mixed, the temperature of the cylinder contents should be maintained above the critical temperature at which CO_2 can liquify and separate.

8.2 *New Cylinders of Gas*—Before using new cylinders of CO/CO_2 reducing gas, mix the contents according to the gas manufacturer's recommendations. To assure that the gas remains mixed, maintain the temperature of the cylinder above the critical temperature at which CO_2 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.

7. Preparation of Ash

7.1 Use coal or coke passing a 250-μm (No. 60) sieve prepared in accordance with Method D2013, to obtain the ash by incineration in a well-ventilated muffle furnace. The quantity of coal or coke required will vary with the ash content; usually 3 to 5 g of ash will be sufficient for cones for several check determinations, if necessary. Spread out the coal or coke in a layer not over 6.4 mm [¼ in.] in depth in a fireclay or porcelain roasting dish. Place the dish in the cold muffle or on the hearth at a low temperature and gradually heat to redness at such a rate as to avoid mechanical loss from too rapid expulsion of volatile matter (Note 3). Complete the conversion to ash at a temperature of 800 to 900°C [1470 to 1650°F]. Transfer the ash to an agate mortar (Note 4) and grind it so that it will pass a 75-μm (No. 200) sieve. Then spread the ash in a thin layer in a fireclay, silica, or porcelain dish and ignite it in a stream of oxygen for 1½ h at 800 to 850°C [1470 to 1560°F] to ensure complete and uniform oxidation of the ash. Any tube or muffle-type furnace which, when supplied with an oxygen flow of not less than one furnace volume in 5 min, will maintain a highly oxidizing atmosphere and will be suitable.

NOTE 3—A heating rate conforming to that used for the ash determination specified in Section 7 of Test Method D3174 is satisfactory.
NOTE 4—A mechanical agate mortar grinder will save time where many determinations are made. An iron mortar or pestle must not be used.

9. Preparation of Cones Sample and Test Specimen Preparation

9.1 *Coal and Coke*—Thoroughly mix the ignited ash in a mechanical mixerPrepare the analysis sample in accordance with Practice D2013or on a for coal or Practice D346 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.for coke by pulverizing the material to pass a 250 μm (No. 60) U.S.A. standard sieve.

9.2 *Laboratory Ashing of Coal or Coke Analysis Sample*—Take sufficient ash for Prepare the ash from a thoroughly mixed analysis sample of coal or coke (see 9.1the number of cones desired from various parts of the bulk ash. Moisten). To facilitate the ashing process, spread the coal or coke in a layer about 6 mm (¼the ash with a few drops of a clear, filtered (if necessary) 10 % solution of dextrin containing 0.1 % salicylic acid as a preservative 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 pressure at the base with a suitably pointed spatula. Previous