



Designation: D2639/D2639M – 21a

# Standard Test Method for Plastic Properties of Coal by the Constant-Torque Gieseler Plastometer<sup>1</sup>

This standard is issued under the fixed designation D2639/D2639M; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 1. Scope

1.1 This test method covers a relative measure of the plastic behavior of coal when heated under prescribed conditions. This test method may be used to obtain semiquantitative values of the plastic properties of coals and blends used in carbonization and in other situations where determination of plastic behavior of coals is of practical importance.

1.2 *Units*—The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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>

[D2013/D2013M Practice for Preparing Coal Samples for Analysis](#)

[D2234/D2234M Practice for Collection of a Gross Sample of Coal](#)

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.15 on Metallurgical Properties of Coal and Coke.

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<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.

## 3. Terminology

3.1 *Definitions:*

3.1.1 *breaking,  $v$* —free spinning behavior of coal, either by an abrupt change to rotating at maximum motor speed or by abrupt changes in rotation, which occurs as a result of a molten ball of coal forming around the base of the stirrer, and which makes reporting of the true maximum fluidity of the coal difficult.

3.1.2 *dial division per minute, (DDPM),  $n$* —as used in this test method, the stirrer rotation rate as measured by the accumulated rotations (full or partial) of the stirrer recorded over sequential 1 min periods based upon 100 dial divisions for each complete 360° rotation of the stirrer.

3.1.2.1 *Discussion*—Historically stirrer rotations were recorded on manual plastometers by a simple timed observation of the total rotations at the completion of each sequential whole minute interval as the furnace was ramped over the required temperature range. Modern instruments have the ability to record the accumulated stirrer rotations at any given point during the test and as such can present the DDPM over any discrete 60 s interval. For the purposes of this empirical standard and in order to ensure consistency between instrumentation, the recorded DDPM shall be taken at the end of each sequential minute interval commencing at the start of the test and ending with the recorded solidification temperature. That is to say, upon regaining the initial temperature of 300 °C, total accumulated stirrer rotations shall be recorded at times 00:01:00, 00:02:00, 00:03:00, 00:04:00, etc. until the solidification temperature is reached.

3.1.3 *initial softening temperature,  $n$* —as used in this test method, the temperature at which the DDPM (see 3.1.2) first reaches a reading of equal to or greater than 1.0.

3.1.4 *jamming,  $v$* —swelling up of coal into the retort tube during the test, which may produce lower fluidity results than expected and can only be noted after visual inspection of the disassembled crucible and retort at the conclusion of the test.

3.1.5 *maximum fluidity,  $n$* —as used in this test method, the measured maximum stirrer rotation rate, in dial divisions per minute.

3.1.6 *maximum fluidity temperature, n*—as used in this test method, the temperature at which stirrer rotation rate reaches a maximum value.

3.1.7 *plastic range, n*—difference between the solidification temperature and the initial softening temperatures.

3.1.8 *solidification temperature, n*—as used in this test method, the temperature at which the DDPM (see 3.1.2) indicates the last stirrer rotation.

3.1.8.1 *Discussion*—Modern instruments have the capability to measure fractional rotations of the stirrer. For the purposes of this test method, the solidification temperature shall be recorded as the temperature corresponding to the DDPM (see 3.1.2) that exhibits the last non-zero reading. Solidification temperature can also be defined as the temperature of the recorded DDPM immediately preceding the first DDPM of zero.

## 4. Summary of Test Method

4.1 The plastometer measures the plastic properties of coals by the use of a constantly applied torque on a stirrer placed in a crucible into which the coal is charged. The crucible is immersed in a bath and the temperature increased uniformly. The rates of movement of the stirrer are recorded in relation to increase in temperature.

## 5. Significance and Use

5.1 Reliable values of the plastic properties of coals are used to predict or explain the behavior of a coal or blends during carbonization or in other processes such as gasification, liquefaction, and combustion.

## 6. Apparatus

6.1 *Gieseler Plastometer*—The apparatus shall consist of the following:

6.1.1 *Retort*—A steel retort consisting of four parts as shown in Fig. 1.

6.1.2 *Retort Crucible*, cylindrical, 21.4 mm  $\pm$  0.08 mm [0.844 in.  $\pm$  0.003 in.] in inside diameter and 35.0 mm [1.38 in.] in depth, with exterior threads for joining the crucible to the crucible cover. The crucible shall have a 2.38 mm  $\pm$  0.02 mm [0.094 in.  $\pm$  0.001 in.] diameter notch in the center of its inside base to serve as a seat for the stirrer.

6.1.3 *Retort Crucible Cover*, with interior threads for joining the crucible cover to the crucible and exterior threads for joining the crucible cover to the barrel. The inside diameter of the hole which accommodates the stirrer is 9.53 mm  $\pm$  0.08 mm [0.375 in.  $\pm$  0.003 in.].

6.1.4 *Barrel*, 121 mm [4.75 in.] long and having an inside diameter of 9.53 mm  $\pm$  0.08 mm [0.375 in.  $\pm$  0.003 in.]. The top end of the barrel shall be 12.7 mm [0.500 in.] in inside diameter to a depth sufficient to allow clearance for the axle of the plastometer head when the apparatus is assembled. A hole, fitted with a tube, shall be provided at the midpoint of the barrel so as to afford exit for the volatile products during a test.

6.1.5 *Steel Stirrer*, provided with a straight shaft 3.96 mm  $\pm$  0.03 mm [0.156 in.  $\pm$  0.001 in.] in diameter and equipped with four rabble arms. The lower end of the stirrer shall have a 60° included angle. The rabble arms on the stirrer

shall be 1.59 mm  $\pm$  0.03 mm [0.0625 in.  $\pm$  0.001 in.] in diameter, 6.35 mm  $\pm$  0.05 mm [0.250 in.  $\pm$  0.002 in.] in length, and shall be placed perpendicular to the shaft at 90° intervals around the shaft and 3.18 mm  $\pm$  0.03 mm [0.125 in.  $\pm$  0.001 in.] apart center to center along the shaft. The middle two rabble arms shall be set at 180° to each other, and likewise, the remaining two arms 180° to each other. The lowest rabble arm shall be set in the shaft to give 1.59 mm [0.0625 in.] clearance between it and the bottom of the crucible when the stirrer is in place. The upper end of the stirrer shall be cut to fit into the slot on the lower end of the axle in the plastometer head.

6.1.6 *Guide Sleeve*, provided near the upper end of the stirrer to guide the latter within the barrel, with a clearance of at least 0.05 mm [0.002 in.].

6.2 *Plastometer Head*—The plastometer head shall consist of a fixed-speed motor connected directly to a hysteresis brake which is capable of adjustment from 29 g·cm to 2.90 g·cm [11.4 g·in. to 114 g·in.] torque. Each complete revolution, or 100 dial division, shall be recorded on a magnetic counter actuated by an electric eye or other suitable method. The torque shall be checked using a drive pulley, transfer pulley, monofilament line, and weights as shown in Fig. 2. Adjust the hysteresis brake torque to lift a 38.00 g  $\pm$  0.1 g [0.0836 lb  $\pm$  0.00022 lb] weight over one full rotation of the slotted axle and not lift a 42.00 g  $\pm$  0.1 g [0.924 lb  $\pm$  0.00022 lb] weight over one full rotation of the slotted axle. The lifted weights include the variable weight of the monofilament line between the transfer pulley and the attached weight. All instruments should be checked in this manner at least once each week.

6.3 *Furnace*—An electrically heated furnace with suitable manual, or automatic, controls shall be provided so that a heating rate of 3.0 °C/min  $\pm$  0.1 °C/min [5.4 °F/min  $\pm$  0.18 °F/min], on an over-all basis, with not more than 3.0 °C  $\pm$  1.0 °C [5.4 °F  $\pm$  1.8 °F] for any given minute can be maintained over a temperature range from 300 °C to 550 °C [572 °F to 1022 °F]. This temperature circuit should be checked and standardized periodically. The furnace shall contain a molten solder bath of approximately 50 % lead and 50 % tin composition. Temperature in the bath shall be measured with a suitable thermocouple contained in an outer protection tube immersed in the bath so that the protection tube touches the outside wall of the crucible, and the measuring junction of the thermocouple is at the same height as the center of the coal charge. A stirrer shall be used to agitate the solder. A suitable furnace is illustrated in Fig. 3.

6.4 *Loading Device*—The loading device shall be provided so that the coal may be packed uniformly in the crucible under a total packing load of 10 kg [22 lb] and designed in such a manner that, after compression, the crucible and its contents can easily be removed from the device without disturbing the contents. An example of the static-weight device is shown in Fig. 4. The use of a static weight of 9 kg [20 lb] together with a drop-weight of 1 kg [2 lb] dropped twelve times from a height of 110 mm [4.5 in.] is permissible as an alternative method of packing. An example of the drop-weight arrangement is shown in Fig. 5. For coals that are difficult to pack, it

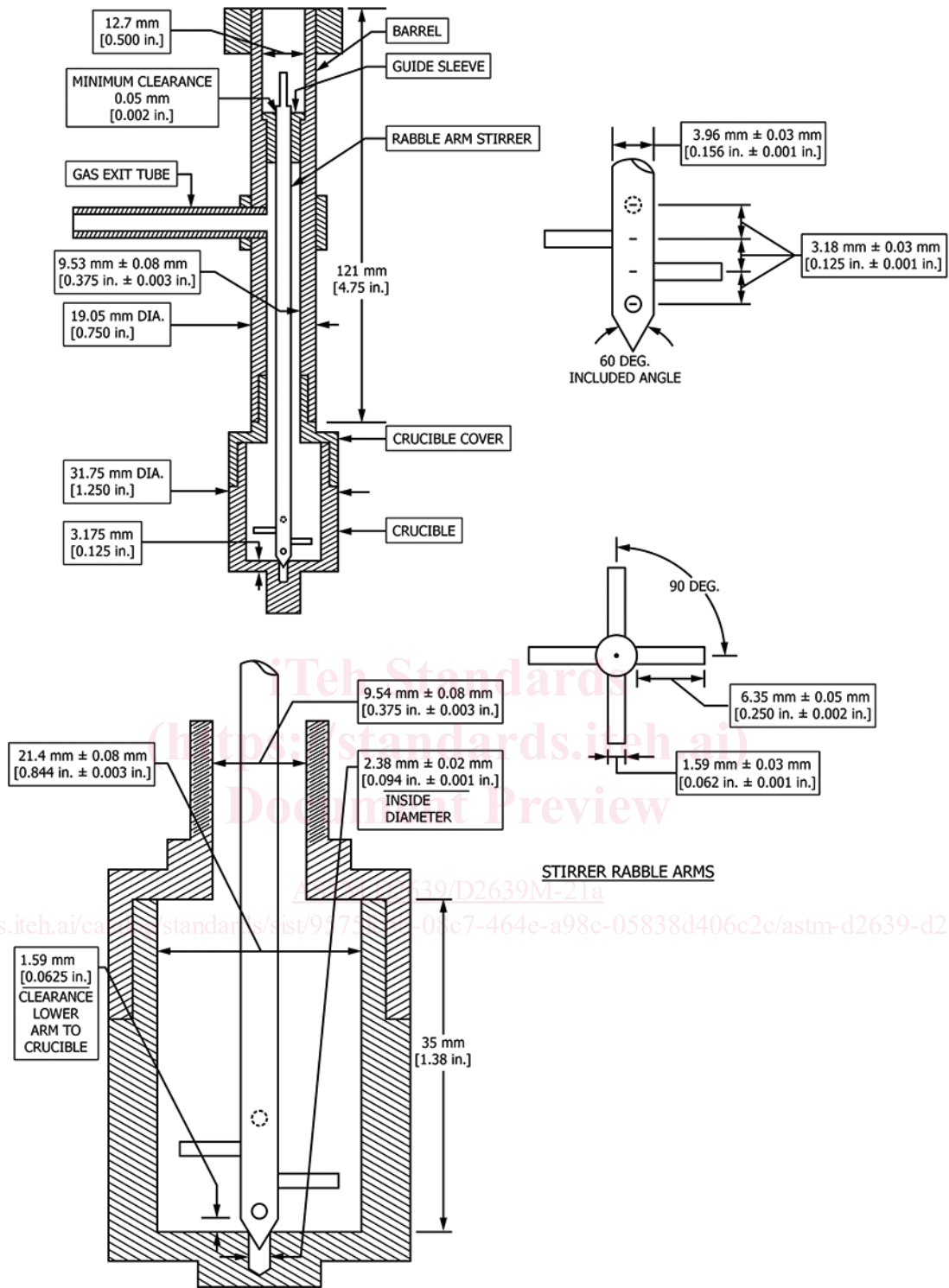


FIG. 1 Retort Assembly

is permissible to add one to three drops of water on the shaft of the stirrer near the surface of the coal before the weight is added.

## 7. Sample

7.1 Collect a representative gross sample of coal in accordance with Practice D2234/D2234M and prepare in accordance

with Practice D2013/D2013M. The 2.36 mm [No. 8] sieve sample from Table 1 in Practice D2013/D2013M shall constitute the laboratory sample.

## 8. Preparation of Sample

8.1 Air dry the sample prior to preparation. Spread the sample on tared pans, weigh, and air dry it at room temperature

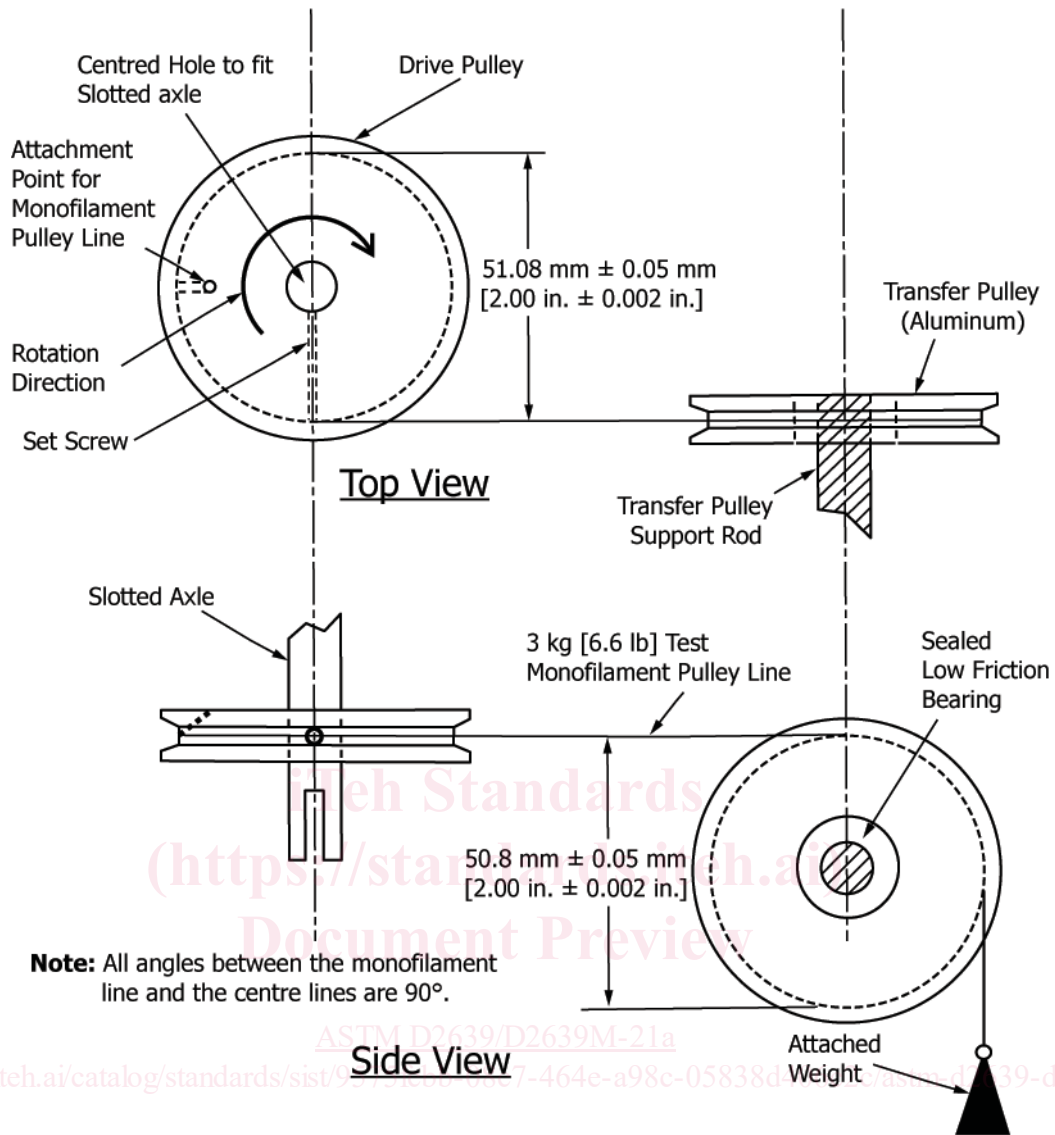


FIG. 2 Pulley Arrangement to Calibrate Torque

or at slightly elevated temperatures not exceeding  $15^\circ\text{C}$  [ $27^\circ\text{F}$ ] above room temperature, until the moisture loss is less than  $0.1\%/\text{h}$ . Drying should not be continued beyond this point so that the plastic properties of the coal are not altered by oxidation. After air drying, reduce the sample by riffing to no less than  $0.5 \text{ kg}$  [ $1 \text{ lb}$ ]. Stage crush this  $0.5 \text{ kg}$  [ $1 \text{ lb}$ ] portion to pass through a  $850 \mu\text{m}$  [No. 20] sieve and reduce to  $250 \text{ g}$  [ $0.55 \text{ lb}$ ] by riffing. Stage crush this  $250 \text{ g}$  [ $0.55 \text{ lb}$ ] portion to pass through a  $425 \mu\text{m}$  [No. 40] sieve and riffle it to  $125 \text{ g}$  [ $0.275 \text{ lb}$ ]. Mix the coal and scoop  $5.0 \text{ g}$  [ $0.01 \text{ lb}$ ] for the test. Seal the excess prepared coal for additional test runs as required. Test the coal for plastic properties within  $8 \text{ h}$  after the sample passes through a  $425 \mu\text{m}$  [No. 40] sieve.

## 9. Procedure

9.1 The hysteresis brake is normally adjusted to  $101.6 \text{ g}\cdot\text{cm} \pm 5.1 \text{ g}\cdot\text{cm}$  [ $40.0 \text{ g}\cdot\text{in.} \pm 2.0 \text{ g}\cdot\text{in.}$ ] torque.

9.2 Mount the crucible, with the stirrer in place, in the loading device as shown in Fig. 4 but with the cage raised. Charge  $5.0 \text{ g}$  [ $0.011 \text{ lb}$ ] of the prepared sample of coal into the crucible. Lower the cage onto the coal and press firmly with the fingers. Rotate the stirrer slightly with the fingers to fill the voids under the rabble arms. Then lower the cage on the coal and apply the  $10 \text{ kg}$  [ $22 \text{ lb}$ ] weight, making sure that the total weight is transmitted to the coal charge. Compress the charge for  $15 \text{ min}$ , after which remove the loading device, taking extreme care not to disturb the position of the stirrer in the coal. If an alternative method of packing is used, use the same care. Screw the barrel on to the plastometer head, making sure that the top of the stirrer fits in the slotted end of the axle.

9.3 Lower the assembled apparatus into the furnace until the bottom of the crucible is immersed to a depth of  $76 \text{ mm}$  [ $3.0 \text{ in}$ ] in the molten solder bath maintained at a temperature of  $300^\circ\text{C}$