



Standard Test Method for Plastic Properties of Coal by the Constant-Torque Gieseler Plastometer¹

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 (ϵ) 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 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 *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

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

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

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

¹ 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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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.

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, a measure of stirrer rotation rate. There are 100 dial divisions for each full 360° rotation of the stirrer. Stirrer rotation rate is the total dial divisions turned by the stirrer in a one minute time period.

3.1.3 *initial softening temperature, n*—as used in this test method, the temperature at which the stirrer rotation rate first reaches 1.0 dial divisions per minute.

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 first zero DDPM is reached after the last stirrer rotation.

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 ± 0.08 mm [0.844 in. ± 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 ± 0.02 mm [0.094 in. ± 0.001 in.]

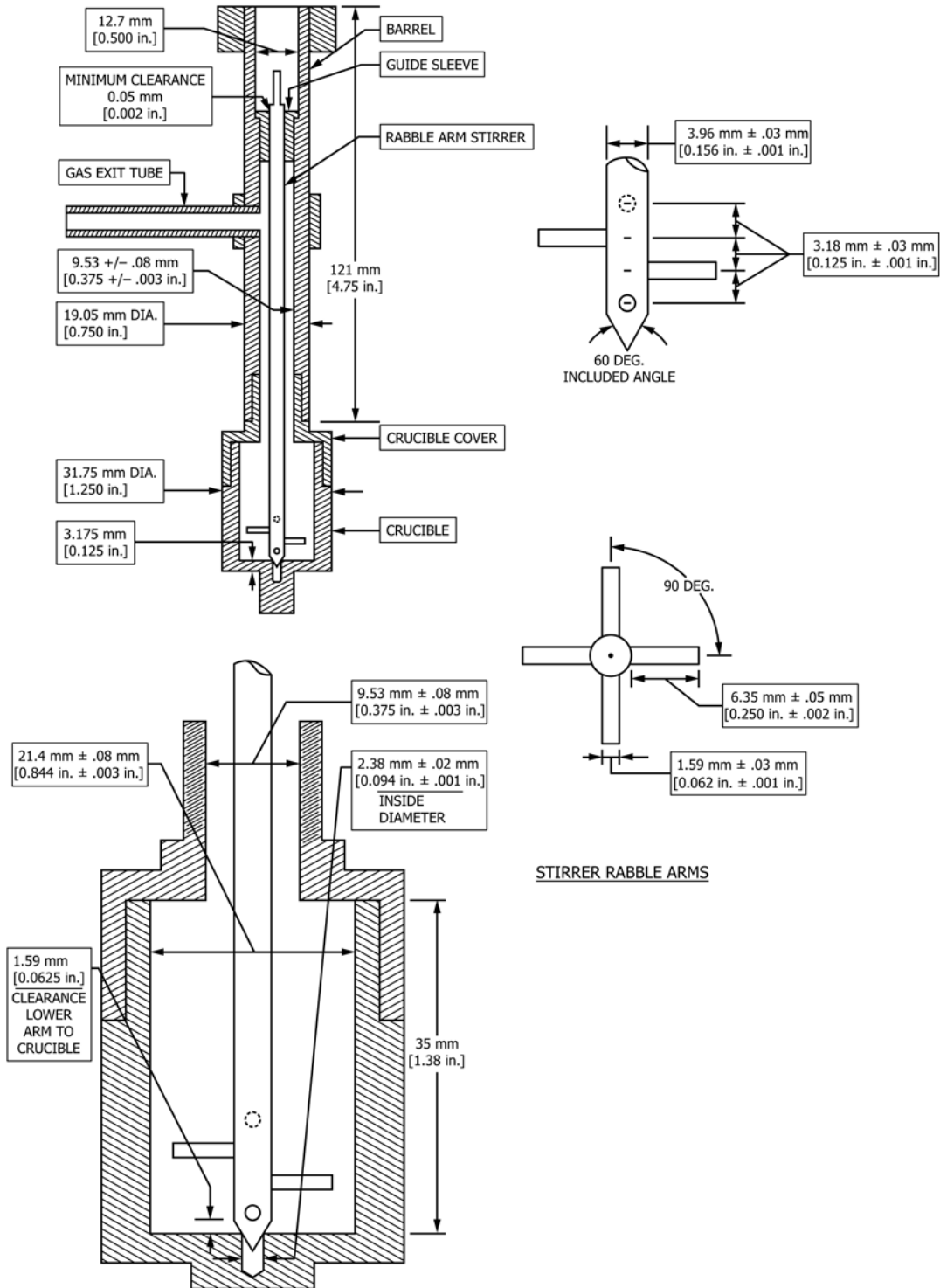


FIG. 1 Retort Assembly

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 in a 6.35 mm [0.250 in.] outside diameter protection tube immersed in the bath so that the tube touches the outside wall of the crucible, and the hot junction of the couple 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. A suitable device is shown in Fig. 4. The use of a static weight of 9 kg [19.8 lb] together with a drop-weight of 1 kg [2.2 lb] dropped twelve times from a height of 114.3 mm [4.5 in.] is permissible as an alternative method of packing. The drop-weight arrangement is shown in Fig. 5. For coals that are difficult to pack, it is permissible to add one to three drops of benzene or toluene 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 or at slightly elevated temperatures not exceeding 15 °C [27 °F] above room temperature, until the moisture loss is less than 0.1 %/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 500 g [1.1 lb]. Stage crush this 500 g [1.1 lb] portion to pass through a 850 μ m (No. 20) sieve and reduce to 250 g [0.55 lb] by riffing. Stage crush this 250 g [0.55 lb] portion to pass through a 425 μ m (No. 40) sieve and riffle it to 125 g [0.275 lb]. Mix the coal and scoop 5.0 g [0.011 lb] for the test. Seal the excess prepared coal for additional test runs as required. Test the coal for plastic properties within eight hours after the sample passes through a 425 μ m (No. 40) sieve.

9. Procedure

9.1 The hysteresis brake is normally adjusted to 101.6 g · cm \pm 5.1 g · cm [40.0 g · in. \pm 2.0 g · 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 g [0.011 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 kg [22 lb] weight, making sure that the total weight is transmitted to the coal charge. Compress the charge for 15 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.

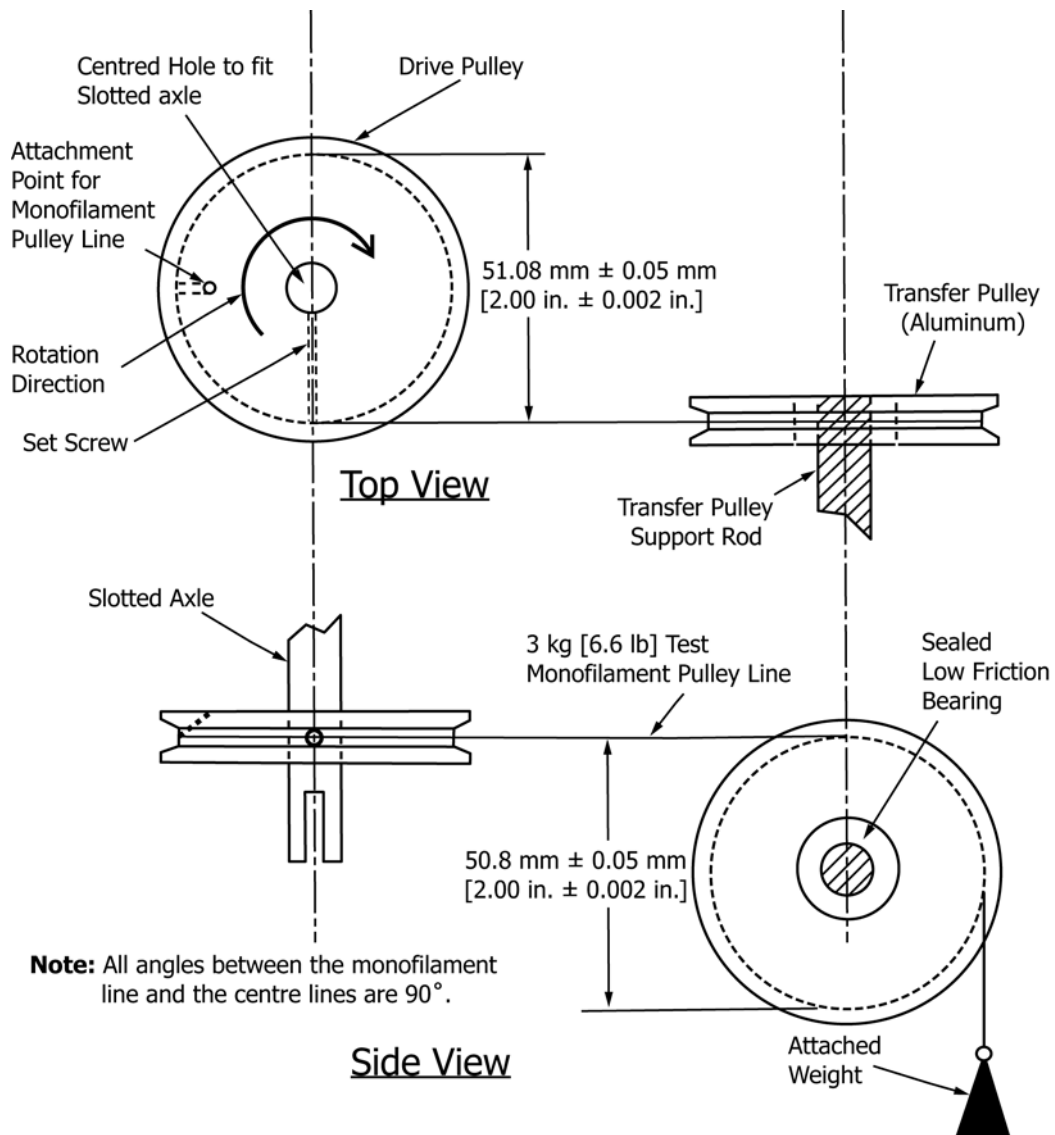


FIG. 2 Pulley Arrangement To Calibrate Torque

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 76.2 mm [3 in.] in the molten solder bath maintained at a temperature of 300 °C [572 °F]. Place the thermocouple in the bath in accordance with 6.3. The heating control shall be such that the bath will reattain the initial temperature in 10 ± 2 min after immersion of the retort. Thereafter, heat the retort at a rate of 3.0 °C/min ± 0.1 °C/min [5.4 °F ± 0.18 °F] on the over-all basis.

9.4 Temperature and stirrer rotation measurements shall be made at 1 min intervals between the initial softening and solidification temperatures for the analyzed coal test sample.

10. Number of Tests

10.1 All tests shall be made in duplicate, and the average values reported.

11. Care of Plastometer

11.1 The dimensions of the rabble arms on the stirrer shall meet the specifications in 6.1.5 and Fig. 1. In addition to thorough cleaning of the stirrer and crucible between tests, it is important that the dimensions of the rabble arms be checked at frequent intervals. The stirrer is to be discarded when any of the rabble arms has a diameter ≤1.47 mm [0.058 in.] or has a ≤6.10 mm [0.24 in.] length.

12. Calculation

12.1 From the measured dial division per minute readings, determine the maximum fluidity of the test sample.

13. Report

13.1 Report the following information: initial softening temperature; maximum fluidity temperature; solidification temperature; plastic range and maximum fluidity.

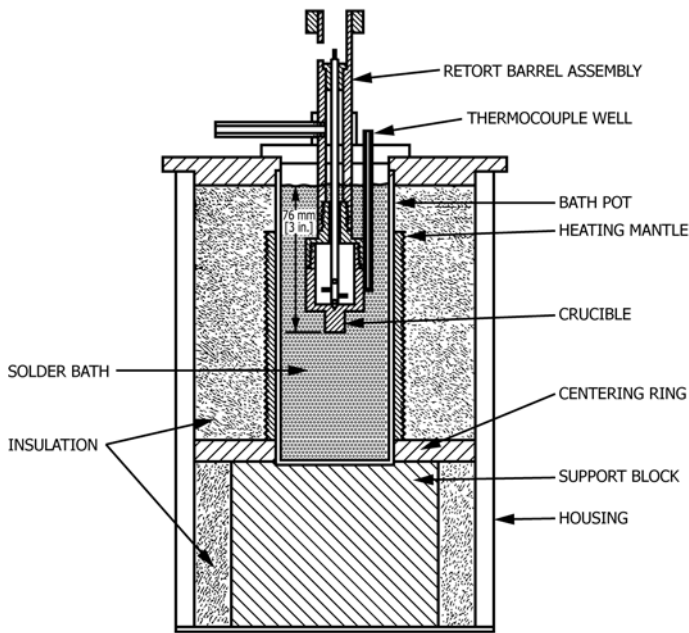


FIG. 3 Furnace Assembly

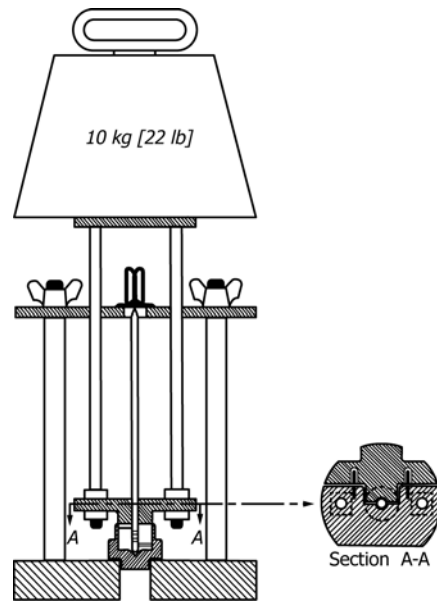


FIG. 5 Loading Device

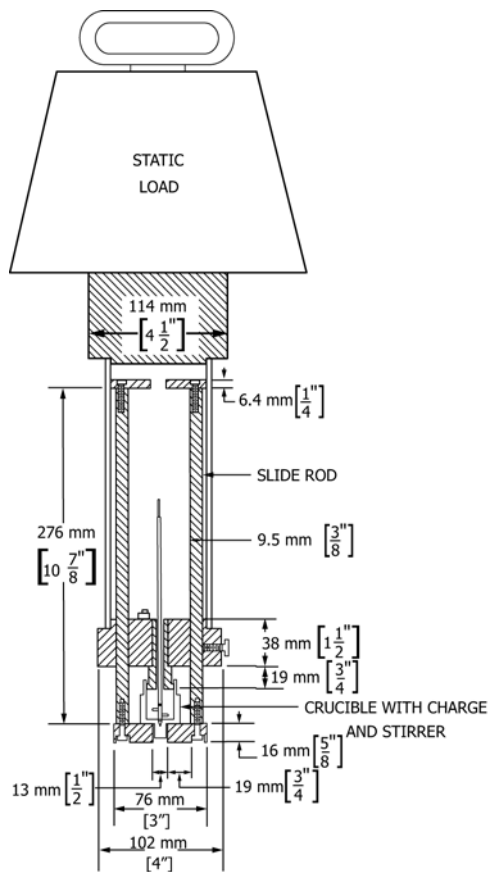


FIG. 4 Loading Device

13.2 Report the arithmetic average of the maximum fluidity for individual determinations that meet the requirements of 14.1.1 as follows:

(a) average maximum fluidity less than 1000 DDPM, report to the nearest 1 DDPM.

(b) average maximum fluidity between 1000 DDPM and 10 000 DDPM, round and report to the nearest 10 DDPM.

(c) average maximum fluidity between 10 000 DDPM and 20 000 DDPM, round and report to the nearest 100 DDPM.

(d) average maximum fluidity between 20 000 DDPM and 30 000 DDPM, round and report to the nearest 1000 DDPM.

(e) average maximum fluidity greater than 30 000 DDPM, report as greater than (>) 30 000 DDPM.

NOTE 1—It is recognized that some plastometers currently in use have the capability to read and record the fluidity to the nearest 0.1 DDPM. For coals which exhibit a maximum fluidity less than 10 DDPM, and where required, it is acceptable to round and report the average maximum fluidity to the nearest 0.1 DDPM.

13.3 If either breaking or jamming was observed, a statement that maximum fluidity cannot be verified due to the phenomenon should be added to the report.

13.4 Example Report:

13.4.1 Individual maximum fluidity results for duplicate determinations are found as 27 536 DDPM and 22 437 DDPM. The average of the duplicate determinations is 24 986 DDPM. Repeatability for the two determinations is ± 2499 DDPM of the average. The repeatability statement in 14.1.1 is not satisfied. A further third determination yields a result of 25 957 DDPM. The average of the first and third determinations, respectively, is 26 746 DDPM. Repeatability for these two determinations is ± 2675 DDPM of the average. The repeatability statement is now satisfied. Rounding the average of the two repeatable determinations to the nearest 1000 DDPM according to 13.2(d) provides a reported result of 27 000 DDPM.

Initial Softening Temperature:	388 °C
Maximum Fluidity Temperature:	440 °C
Solidification Temperature:	490 °C
Plastic Range:	102 °C
Maximum Fluidity:	27 000 DDPM



14. Precision and Bias

14.1 Precision:

14.1.1 *Repeatability*—For duplicate tests carried out at different times in the same laboratory, by the same operator, with the same apparatus, on representative portions taken from the same sample after the last stage of the reduction process, all characteristic temperature points shall agree within 5 °C [9 °F]. Likewise, the maximum fluidity of duplicate tests prior to rounding shall agree within ± 10 % of the arithmetic average of the two tests. If these criteria are not met, a third test shall be run. If there is agreement between two of the three tests, calculate the arithmetic average of the two determinations, round (if required) and report. If agreement between two of the three tests is unsatisfactory, calculate the arithmetic average of the three individual determinations, round (if required) and report.

14.1.2 *Reproducibility*—It is not practical to specify the reproducibility of this test method because numerous round-robin experiments have demonstrated inherent variance of test equipment.

NOTE 2—Round-robin experiments have included both coal and synthetic standards.

14.1.2.1 It has been determined there are no valid statistical data for the establishment of a reproducibility statement for this test method. This test method should, therefore, be used only as an indicator of the plastic properties of coal.

14.2 *Bias*—This test method, including calibration, is an empirical procedure. Therefore, the degree of absolute bias cannot be determined.

15. Keywords

15.1 carbonization; contraction; dial divisions; expansion; fluidity; gieseler; plasticity; rheology

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