Designation: D 2639 – 98

Standard Test Method for Plastic Properties of Coal by the Constant-Torque Gieseler Plastometer[1]

This standard is issued under the fixed designation D 2639; 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.

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 SI units are to be regarded as the standard. The values given in parentheses are for information only.

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:*

D 2013 Method of Preparing Coal Samples for Analysis[2]

D 2234 Practice for Collection of a Gross Sample of Coal[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *dial division per minute, 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 total dial divisions turned by the stirrer in a one minute time period.

3.1.2 *final fluid temperature, n*—as used in this test method, the temperature at which stirrer rotation rate last reaches 1.0 dial divisions per minute.

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

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

3.1.6 *plastic range, n*—difference between the final fluid and the initial softening temperatures.

3.1.7 *solidification temperature, n*—as used in this test method, the temperature at which the last stirrer rotation is observed.

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 ± 0.08 mm (0.844 ± 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- ± 0.02-mm (0.094- ± 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 ± 0.08 mm (0.375 ± 0.003 in.).

6.1.4 *Barrel*, 121 mm (4.75 in.) long and having an inside diameter of 9.53 ± 0.08 mm (0.375 ± 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

[1] This test method is under the jurisdiction of ASTM Committee D-5 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] *Annual Book of ASTM Standards*, Vol 05.05.

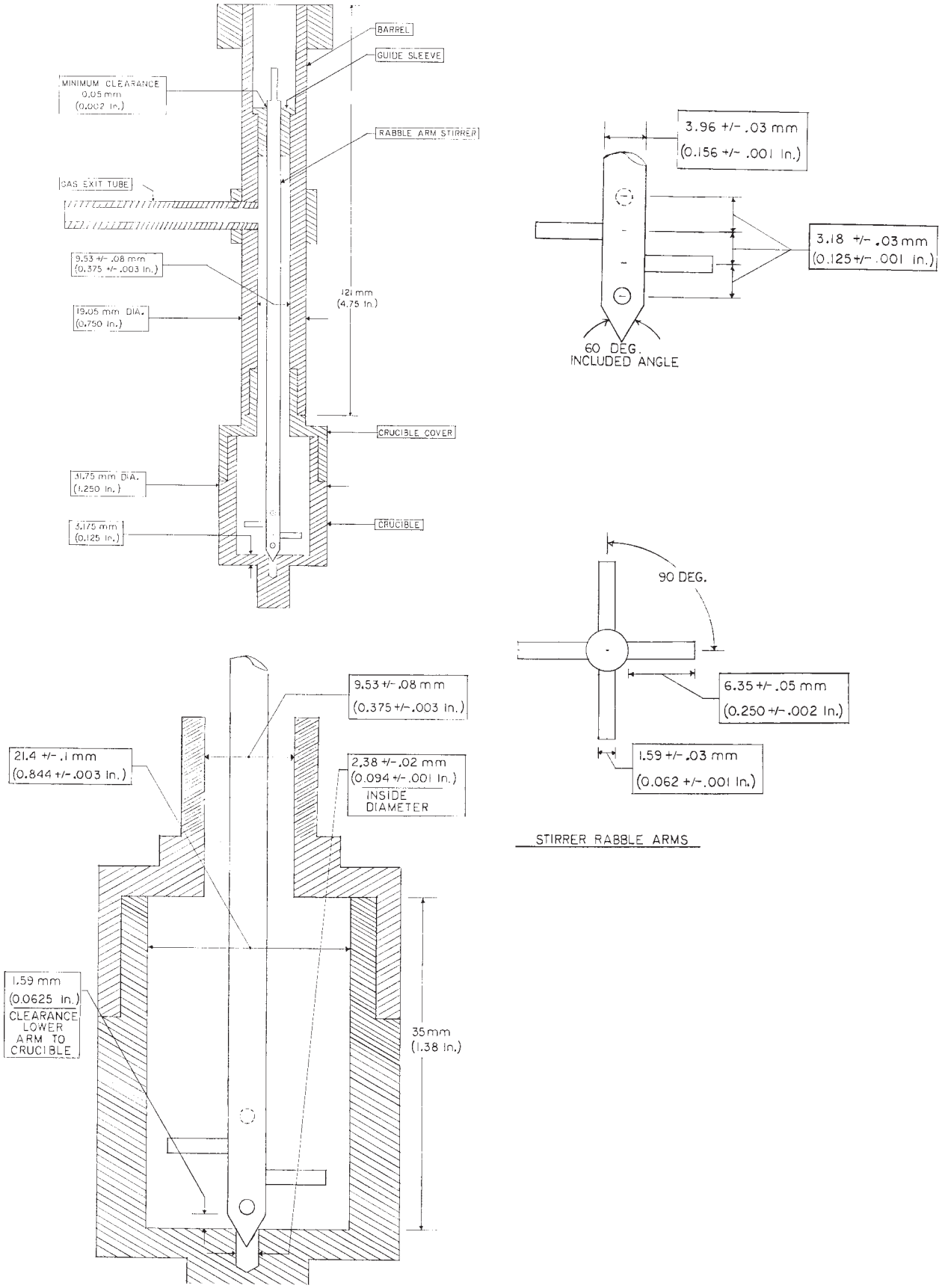

FIG. 1 Retort Assembly

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 ± 0.03 mm (0.156 ± 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 ± 0.03 mm (0.0625 ± 0.001 in.) in diameter, 6.35 ± 0.05 mm (0.250 ± 0.002 in.) in length, and shall be placed perpendicular to the shaft at 90° intervals around the shaft and 3.18 ± 0.03 mm (0.125 ± 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- to 2.90-g · cm (11.4- 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- ± 0.1-g weight over one full

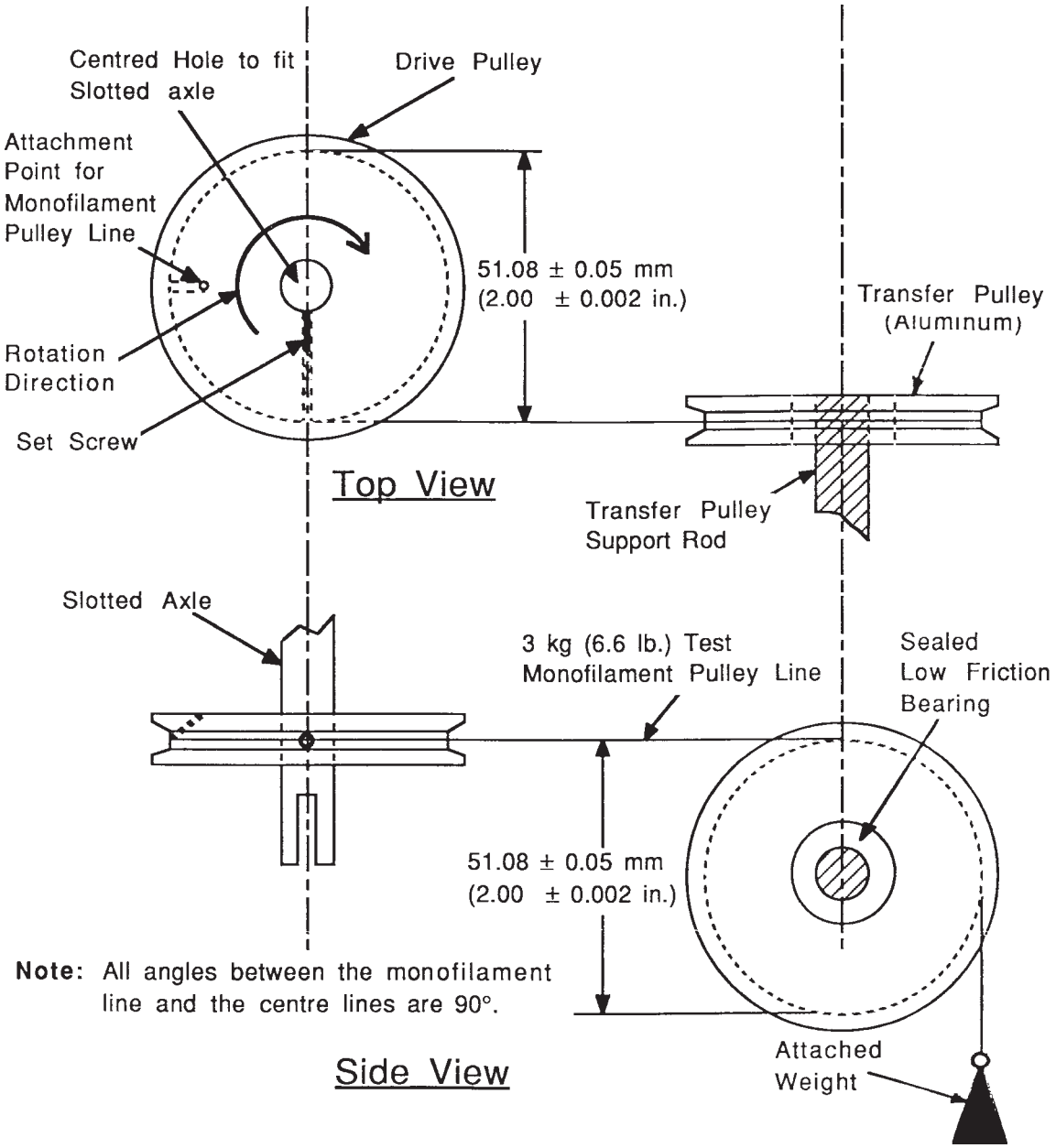

FIG. 2 Pulley Arrangement To Calibrate Torque

rotation of the slotted axle and not lift a 42.00- ± 0.1-g 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 ± 0.1°C/min, on an over-all basis, with not more than 3.0 ± 1.0°C for any given minute can be maintained over a temperature range from 300 to 550°C. 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 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 together with a drop-weight of 1 kg 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 Test Methods D 2234 and prepare in accordance with Method D 2013. Approximately 4 kg of coal crushed to pass a 4.75-mm (No. 4) sieve shall constitute the laboratory sample.

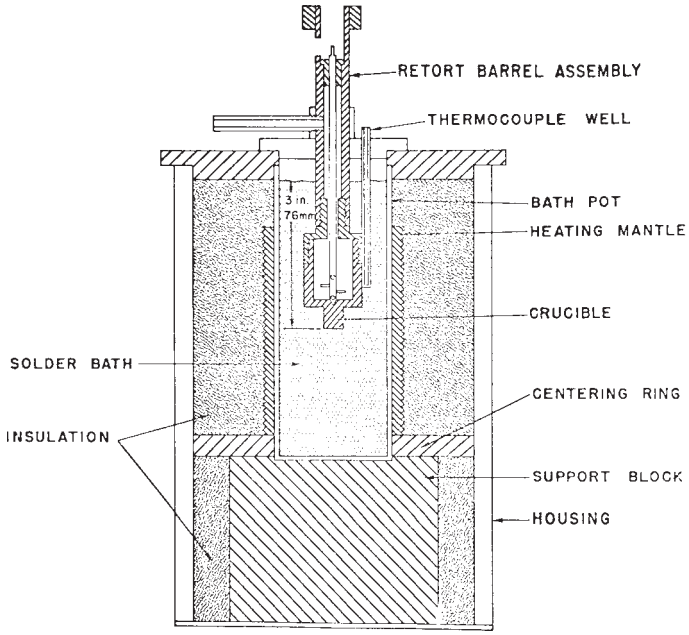

FIG. 3 Furnace Assembly

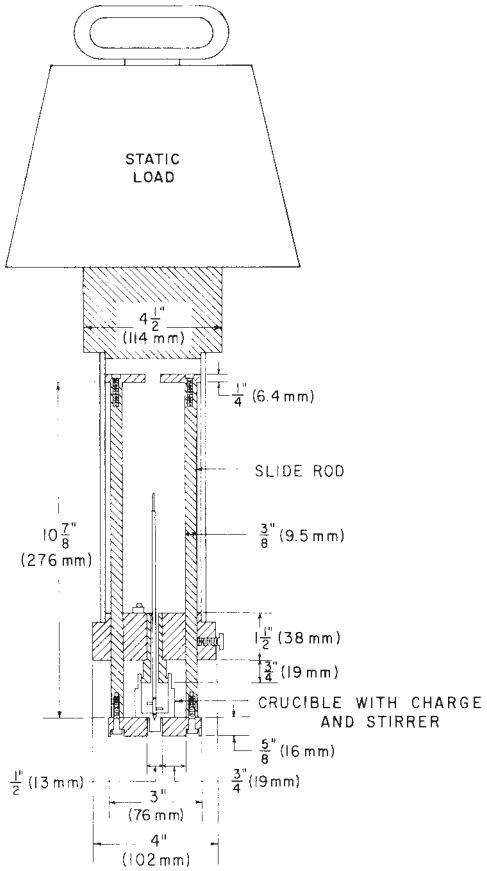

FIG. 4 Loading Device

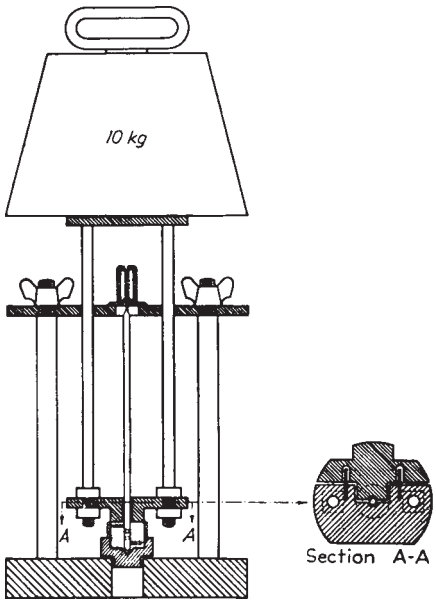

FIG. 5 Loading Device

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 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, grind the 4-kg laboratory sample to pass an 850-µm (No. 20) sieve and reduce it by riffling to about 500 g. Quarter, preferably by riffling, this 500-g portion and stage-crush one quarter to pass a 425-µm (No. 40) sieve in such a manner as to minimize production of fines. Test the coal for plastic properties as soon as possible after preparing the sample passing the 425-µm sieve.

9. Procedure

9.1 The hysteresis brake is normally adjusted to 101.6- ± 5.1-g · cm (40.0- ± 2.0-g · in.) torque.

9.2 Mount the crucible, with the stirrer in place, in the tk;2loading device as shown in Fig. 3 but with the cage raised. Charge 5.0 g 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 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. 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. 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 ± 0.1°C/min 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 5.1.4 and Fig. 2. 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. Calculate the logarithm (Base 10) of the maximum fluidity if maximum fluidity >1 dial division per minute.

13. Report

13.1 Report the following information:

13.1.1 Characteristic temperatures: initial softening; maximum fluidity; final fluid; solidification; and plastic range.

13.1.2 Maximum fluidity and logarithm (Base 10) maximum fluidity to one decimal place if maximum fluidity >1 dial division per minute.

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 should agree within 5°C. Likewise, maximum rates of drum dial movement of duplicate tests should agree within ±10 % of the arithmetic average of the two tests. If these criteria are not met, the results should be discarded and a second set of tests run. If agreement between tests in the second set is unsatisfactory, these results should be discarded also, and a third set of tests run. If agreement between tests in the third set is unsatisfactory, the average of the six individual tests should be reported.

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 1—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; geisler; plasticity; rheology