



Designation: D 5515 – 97

## Standard Test Method for Determination of the Swelling Properties of Bituminous Coal Using a Dilatometer<sup>1</sup>

This standard is issued under the fixed designation D 5515; 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.

### INTRODUCTION

The principle of this test method is that the final volume of char obtained at the conclusion of a standard dilatation test is dependent on the mass of coal in the coal pencil and on the radius of the retort tube. This test method incorporates a procedure which: determines the mass of air-dried coal in the coal pencil; provides a means to measure the average retort tube radii; and employs a means to report coal expansion on an air dried coal weight basis.

Other test methods used to determine the swelling properties of bituminous coals include the Ruhr (ISO 8264) and Audibert-Arnu (ISO 349) International Standard Organization (ISO) test methods. However these two ISO test methods provide consistently different values for percent dilatation and percent contraction. Percent contraction and dilatation values obtained using the Audibert-Arnu test method are higher and lower respectively than those obtained using the Ruhr test method. These differences have been attributed to trimming the length of the coal pencil from different ends. The Audibert-Arnu test method specifies that the wider end of the coal pencil be trimmed while the Ruhr test method specifies that the narrower end of the coal pencil be trimmed.

### 1. Scope

1.1 This test method specifies a procedure for the measurement of the swelling of bituminous coal using a dilatometer.

1.2 The test method is limited in applicability to those coals which have a free swelling index  $\geq 1$  as determined in accordance with Test Method D 720.

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.*

1.4 The values stated in SI units (IEEE/ASTM SI-10) are to be regarded as standard.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 720 Test Method for Free-Swelling Index of Coal<sup>2</sup>
- D 2013 Method of Preparing Coal Samples for Analysis<sup>2</sup>
- D 2234 Practice for Collection of a Gross Sample of Coal<sup>2</sup>
- IEEE/ASTM SI-10 Standard for Use of the International

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<sup>2</sup> Annual Book of ASTM Standards, Vol 05.05.

System of Units (SI): The Modern Metric System<sup>3</sup>

#### 2.2 International Standardization Organization (ISO) Standards:

- ISO 349 Hard Coal-Audibert-Arnu Dilatometer Test
- ISO 8264 Hard Coal—Determination of the Swelling Properties Using a Dilatometer

### 3. Terminology

3.1 *Abbreviations:* basement level reference mark height, BLRM<sub>ht</sub>, the char height as measured using the BLRM, after removing the piston/retort assembly as a unit after completion of the test, expressed in millimetres.

3.2 *coal pencil*, a 60-mm-long test specimen formed by compression in a mold from coal which has been pulverized to pass a 250- $\mu\text{m}$  (No. 60) sieve.

3.3 *dry coal pencil mass*,  $M_{\text{dry}}$ , the calculated mass of the trimmed 60-mm-long coal pencil corrected for added water, expressed in grams.

3.4 *equivalent percent dilatation for 2.50 g of air dried coal*, %D<sub>2.50</sub>, the calculated percent expansion for a 2.50-g, unmoistened, 60-mm-long, coal pencil corrected for average tube radii, expressed as a percentage.

3.5 *maximum contraction temperature*,  $T_2$ , the temperature at which the coal pencil starts swelling, expressed in Celsius. For coals which exhibit contraction only,  $T_2$  is the temperature

<sup>3</sup> Annual Book of ASTM Standards, Vol 14.02.

at which the coal pencil reaches its minimum (see Fig. 1). For coals that exhibit contraction only and are still contracting at 500°C, T2 will be reported as *taken at 500°C* (see Fig. 1d).

3.6 *maximum dilatation temperature, T3*, the temperature at which the coal pencil first reaches a maximum height after swelling, expressed in Celsius (see Fig. 1).

3.7 *percent contraction, %C*, the minimum recorded height of char expressed as a percentage, based on an initial coal pencil height of 60 mm (see Fig. 1).

3.8 *percent dilatation, %D*, the maximum recorded height of char expressed as a percentage, based on an initial coal pencil height of 60 mm (see Fig. 1).

3.9 *softening temperature, T1*, the temperature at which the height of the coal pencil contracts 1.0 % (0.6 mm) from the highest recorded initial pencil height, expressed in Celsius (see Fig. 1).

3.10 *wet coal pencil mass, M<sub>wet</sub>*, the measured mass of a trimmed 60-mm-long coal pencil weight, expressed in grams.

**4. Summary of Test Method**

4.1 The test involves preparing a coal pencil and determining the changes of the coal pencil height in a retort tube during a prescribed heating cycle.

**5. Significance and Use**

5.1 Values of the dilatation properties of coals may be 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 *Dilatometer apparatus*, a typical arrangement of the dilatometer apparatus is shown in Fig. 2. The apparatus consists of the following:

6.1.1 *Electric Furnace*—The dilatometer furnace has a core consisting of a 65-mm-diameter cylindrical block of aluminum bronze, resistant to oxidation and having a sufficiently high melting point. The length of the furnace core can vary from 400 to 460 mm depending on the length of the retort tubes used. The block has three symmetrically placed 15-mm-diameter bored holes capable of accepting three retort tubes. The core is heated electrically by insulated resistance windings capable of being controlled at a temperature ramp rate of  $3.0 \pm 0.1^\circ\text{C}$  per minute from within 7 min of the time a test is started to a final temperature of 520°C. The furnace shall perform in accordance with the specifications outlined in 8.2. The thermocouple used for temperature control is situated in a retort tube placed in the third hole of the core. The thermocouple tip is placed in contact with the retort tube wall 60 mm above the bottom of the retort tube plug.

6.1.2 *System for Measuring Piston Movement and Temperature*—The system shall be capable of measuring the linear displacement of the piston to the nearest 1.0 mm and providing a correlation of displacement with temperature.

6.1.3 *Retort Tube and Pistons*—The retort tube consists of a seamless tube of steel, with an internal diameter of  $8.00 \pm 0.05$  mm and an external diameter of  $14.50 \pm 0.05$  mm. It shall have a support collar at the top and be threaded to accept a gastight, threaded plug at its base. The retort tube shall have a minimum

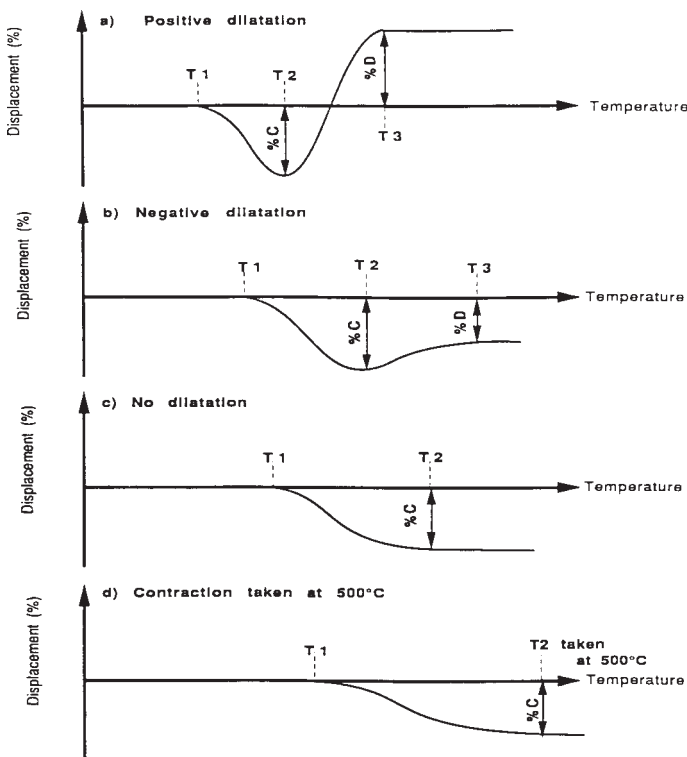
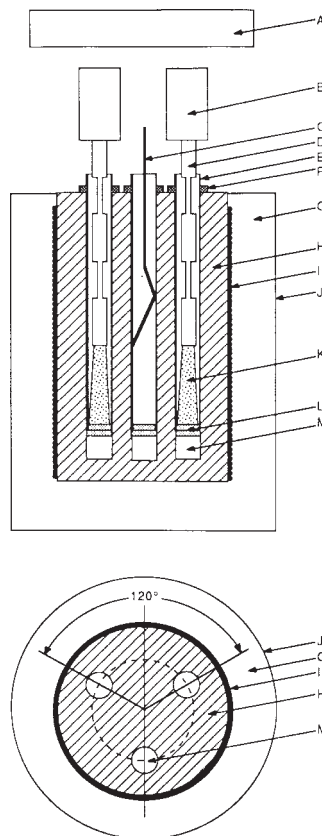


FIG. 1 Types of Dilatation Curves



- A - Piston movement and temperature recording device
- B - Mechanical recording or transducer assembly
- C - Sheathed thermocouple
- D - Piston
- E - Retort tube
- F - Piston collar
- G - Refractory insulation
- H - Furnace core
- I - Heating elements
- J - Furnace casing
- K - Test specimen
- L - Threaded plug
- M - Holes for retort tube (15.0 ± 0.1 mm diameter on hole centre radius of 20.0

FIG. 2 Typical Dilatometer Apparatus

length of 345 mm and be supported only by its collar when placed in the furnace. The retort tube shall be discarded when its internal diameter exceeds 8.15 mm.

6.1.3.1 The piston shall be machined from a steel rod to a finished diameter of  $7.80 \pm 0.05$  mm. The piston shall be of sufficient length to permit the mechanical recording or transducer assembly to record a coal pencil height of 30 mm. The piston and mechanical recording or transducer assembly shall weigh  $150 \pm 10$  g. The piston shall be discarded when its diameter is less than 7.65 mm.

6.1.3.2 The piston and retort tube shall be marked and used as a matched set and require identification imprinted on both the retort tube and its matched piston. A line, called the Basement Level Reference Mark (BLRM), shall be imprinted on the piston, so when assembled with its matched retort tube, with plug in place, the BLRM is even with the top of the retort tube. The length from the bottom of the piston to the BLRM is the length of the retort tube bore with plug in place.

6.2 *Mold and Accessories*—The mold and accessories shall be capable of producing a  $60.0 \pm 0.5$ -mm-long coal pencil. The mold shall have a  $70.0 \pm 0.1$ -mm inside bore length with a taper of 1:50. The narrow end of the bore shall have a diameter of  $6.00 \pm 0.01$  mm. Use a trimming block to facilitate the trimming of the coal pencil to length and the subsequent weighing of the trimmed pencil (see Note 1). The portion of the trimming block holding the pencil shall not weigh more than 155 g.

NOTE 1—The coal pencil may be trimmed to length from either end.

6.3 *Cleaning Implements (Recommended)*—The cleaning implements for the retort tube consist of an approximately 7.95-mm-diameter reamer and a 9-mm bronze wire rifle brush with suitable attachments to permit cleaning of the full length of the retort tube. A circular wire brush, attached to a bench top mounted grinder, is recommended for the cleaning of the pistons.

6.4 *Balance*—The balance shall have a weighing range of 160.00 g with 0.01-g readability.

## 7. Preparation of Sample

7.1 Collect a gross sample in accordance with the requirements of Test Method D 2234.

7.2 The analysis sample shall consist of a minimum of 50 g of coal pulverized to 250- $\mu$ m (No. 60) sieve in accordance with Test Method D 2013. Perform the dilatation analysis on the coal sample passing 250- $\mu$ m (No. 60) sieve within five days after reduction to 250- $\mu$ m (No. 60) sieve.

## 8. Calibration

8.1 For all systems not using a mechanical pen/chart, calibrate the recorder of piston height when there is a difference of 3.0 mm or more between the final char heights as calculated from the chart readout and those directly measured using the BLRM for two consecutive determinations.

8.2 The furnace, while being ramped at 3°C per minute, must meet the following temperature criteria with the empty retort tubes placed in Bores 1 and 2 of the furnace and all temperature measurements made with the thermocouple(s) in contact with the retort tube wall(s): temperatures measured in

Bores 1 and 2 must be  $\pm 3.0^\circ\text{C}$  measured at the same height for 45-mm intervals encompassing the bottom 180 mm; temperatures between the control thermocouple placed in its usual position and those of Bores 1 and 2 measured at 45-mm intervals shall be  $\pm 3.0^\circ\text{C}$  for the bottom 135 mm and  $\pm 6^\circ\text{C}$  for the next 45 mm. The furnace temperature profile shall be calibrated every twelve months.

8.3 The dilatometer temperature readout of the measuring thermocouple shall be checked every four months by comparison with a digital thermometer which is calibrated for the same type of thermocouple. If the temperature difference is greater than 3°C, then the dilatometer furnace temperature readout requires recalibration using a temperature calibrator.

8.4 Calibrate the retort tubes, as described in Annex A1, every four months.

## 9. Preparation of the Coal Pencil

9.1 All weights shall be recorded to the nearest 0.01 g. Care must be taken to avoid sample loss and the following steps must be performed without a break.

9.1.1 *Preparation of Homogeneous Coal Sample and Water Mix*—The amount of water in the mix shall be  $\leq 11\%$  on a weight basis.

9.1.1.1 Weigh a mixing vessel. Record the mass as M1.

9.1.1.2 Add approximately 2 g of coal to the preweighed mixing vessel; distribute the coal evenly over the bottom of the mixing vessel. Record the mass as M2.

9.1.1.3 Add approximately 1 mL of distilled water to the coal. Record the mass as M3.

9.1.1.4 Mix the coal and water together using a metal spatula to form a homogeneous paste. Care must be taken to avoid loss of sample.

9.1.1.5 Add another increment of approximately 8 g of coal. Record the mass as M4. Mix to homogeneity using a metal spatula.

9.2 *Construction of the Coal Pencil:*

9.2.1 If a mold release agent has been applied to the bore of the pencil mold, then excess release agent must be removed by forcing a wad of tissue through the bore of the mold before adding the moistened coal.

9.2.2 Add not less than ten increments of the moistened coal to a mold and compact.

9.2.3 Press the coal pencil out of the mold using equipment provided. Gently place the pencil into a trimming block and trim the pencil to a 60-mm length. Discard the trimmed off portion of the pencil. Record the mass of the trimmed coal pencil and trimming block as M5.

9.3 Transfer the trimmed coal pencil into the top of the horizontally placed retort tube. The coal pencil is gently pushed to the bottom of the retort tube with the matched piston. Check that the matched piston slides freely inside the retort tube. Record the mass of the trimming block and any fragments of the trimmed pencil not transferred into the retort tube as M6. To determine the accurate mass of coal in the coal pencil, it is essential that any untransferred fragments of the coal pencil are weighed along with the trimming block.

## 10. Procedure

10.1 Clean the dilatometer retort and piston assembly. Firmly attach the retort tube plugs to the retort tubes. Check that the matched piston slides freely in the retort tube.

10.2 Prepare the coal pencils and load them into the retort tubes as described in Section 9.

10.3 Load both retort tube and piston assemblies, charged with trimmed coal pencils, into the dilatometer furnace which is thermally stabilized at its 315°C setpoint. Start the temperature control program immediately after loading is completed.

10.4 Attach the mechanism used to measure piston height and adjust the piston height reading to  $0 \pm 5\%$  D.

10.5 Terminate the test when no movement of the piston can be detected for 5 min after completion of the dilatation process. In the instance of no dilatation, terminate the test when the furnace temperature reaches 500°C. Reset the furnace to  $315 \pm 5^\circ\text{C}$ . Remove the piston height-measuring mechanism. Remove the retort tube and piston assemblies as a unit. Measure and record the BLRM heights for each piston/retort tube assembly. Allow the assembly to cool to room temperature before cleaning.

10.6 Determine and record the softening (T1), maximum contraction (T2), maximum dilatation (T3) temperatures, percent contraction (%C), and percent dilatation (%D) as defined in Section 3, from the dilatation analysis temperature and height data.

## 11. Number of Tests

11.1 All tests shall be made in duplicate.

## 12. Calculation

12.1 Calculate the percent water added to the coal before pencil making,  $\%_{\text{H}_2\text{O}}$ , as follows:

where:

- $\%_{\text{H}_2\text{O}}$  = amount of water added to the coal, %;
- M3 = mass of the mixing vessel plus first coal increment plus water, g;
- M2 = mass of the mixing vessel plus first coal increment, g;
- M1 = mass of the mixing vessel, g; and
- M4 = mass of the mixing vessel plus first coal increment plus water plus last coal increment, g.

12.2 Calculate the mass of the prepared coal pencil,  $M_{\text{wet}}$ , as follows:

$$M_{\text{wet}} = (M5 - M6) \quad (1)$$

where:

- $M_{\text{wet}}$  = mass of the prepared coal pencil, g;
- M5 = mass of the trimmed coal pencil and trimming block, g; and
- M6 = mass of the trimming block and any untransferred portion of the coal pencil, g.

12.3 Calculate the mass of air-dried coal in the coal pencil,  $M_{\text{dry}}$ , as follows:

$$M_{\text{dry}} = M_{\text{wet}} - (M_{\text{wet}} \times \%_{\text{H}_2\text{O}} \times 0.01) \quad (2)$$

where:

- $M_{\text{dry}}$  = mass of air-dried coal in the coal pencil, g;

- $M_{\text{wet}}$  = mass of the prepared coal pencil calculated in 12.2; and
- $\%_{\text{H}_2\text{O}}$  = amount of water added to the coal calculated in 12.1.

12.4 Calculate the char height,  $H_c$ , as follows:

$$H_c = \%D \times 0.6 + 60 \quad (3)$$

where:

- $H_c$  = calculated char height, mm;
- $\%D$  = dilatation for a 60-mm-long trimmed coal pencil, %;
- 0.6 = the factor used to convert  $\%D$  to a height, mm/%; and
- 60 = the baseline height of the coal pencil, mm.

12.5 Calculate the char height for 2.50 g of air-dried coal,  $H_{2.50}$ , as follows:

$$H_{2.50} = (H_c/M_{\text{dry}}) \times 2.50 \quad (4)$$

where:

- $H_{2.50}$  = char height for 2.50 g of air-dried coal, mm/g;
- $H_c$  = char height calculated in 12.4; and
- $M_{\text{dry}}$  = mass of air-dried coal in the coal pencil calculated in 12.3.

12.6 Calculate the char volume for 2.50 g of air-dried coal,  $V_{2.50}$ , as follows:

$$V_{2.50} = \pi \times R_{\text{av}}^2 \times H_{2.50} \quad (5)$$

where:

- $V_{2.50}$  = char volume for 2.50 g of air-dried coal,  $\text{mm}^3$ ;
- $R_{\text{av}}$  = radius of the retort tube as determined in Annex A1, mm; and
- $H_{2.50}$  = char height for 2.50 g of air-dried coal calculated in 12.5.

12.7 Calculate the height 2.50 g of air-dried coal would occupy in a retort tube of 4.00-mm radius,  $H_{R4}$ , as follows:

$$H_{R4} = V_{2.50}/16/\pi \quad (6)$$

where:

- $H_{R4}$  = height 2.50 g of air-dried coal would occupy in 4.00-mm radius retort tube, mm and
- $V_{2.50}$  = char volume for 2.50 g of air-dried coal calculated in 12.6.

12.8 Calculate percent dilatation for 2.50 g of air-dried coal with retort tube radius corrected to 4.00 mm,  $\%D_{2.5}$ , as follows:

$$\%D_{2.5} = 100 \times (H_{R4} - 60)/60 \quad (7)$$

where:

- $\%D_{2.5}$  = percent dilatation for 2.50 g of air-dried coal and 4.00 mm retort tube radius, % and
- $H_{R4}$  = height 2.50 g of air-dried coal would occupy in 4.00-mm radius retort tube calculated in 12.7.

## 13. Report

13.1 Report softening temperature as T1, maximum contraction temperature as T2, maximum dilatation temperature as T3, percent dilatation as  $\%D$ , and percent contraction as  $\%C$  as determined in 10.5. Report percent water added as  $\%_{\text{H}_2\text{O}}$  as determined in 12.1, dry coal pencil mass as  $M_{\text{dry}}$  as determined

in Section 12.3, and equivalent percent dilatation for 2.50 g of dry coal corrected to a 4.00-mm retort tube radius as %  $D_{2.50}$  as determined in 12.8.

13.2 Temperature and percent expansion and contraction values of an acceptable duplicate analysis shall be averaged and reported to the nearest whole number.

13.3 Dry coal pencil weight of an acceptable duplicate analysis shall be averaged and reported to the nearest 0.01 g.

13.4 Percent water added of an acceptable duplicate analysis shall be reported to the nearest 0.1 g.

#### 14. Precision and Bias

14.1 *Precision*—The relative precision of this test method for the measurement of the swelling of bituminous coal was calculated from data obtained from coals with dilatation ranges shown in Table 1.

**TABLE 1 Temperatures and Limits for Repeatability and Reproducibility for the Swelling Properties of Bituminous Coals**

Measured Parameter	Range	Repeatability, $I(r)$	Reproducibility, $I(R)$
T1	360-440°C	6	20
T2	415-475°C	6	20
T3	465-495°C	6	20
% $D_{2.50}$	<100 %	7	17
% $D_{2.50}$	100-200 %	16	35

14.2 *Repeatability*—The difference in absolute value between two consecutive test results, carried out on the same analysis sample, in the same laboratory by the same operator, using the same apparatus, should not exceed the repeatability limit  $I(r)$  more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability limit, there is reason to question one, or both, of these test results. The repeatability limits for the parameters determined by this method are described in Table 1.

14.3 *Reproducibility*—The difference in absolute value of replicate determinations carried out in different laboratories, on representative samples prepared from the same bulk sample after the last stage of reduction, should not exceed the reproducibility limit  $I(R)$  more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the reproducibility limit, there is reason to question one, or both, of these test results. The reproducibility limits for the parameters determined by this method are described in Table 1.

14.4 *Bias*—Since no suitable certified reference materials for selling properties of bituminous coal using a dilatometer are currently available, no statement on absolute bias can be made for this test method.

#### 15. Keywords

15.1 bituminous coal; dilatometer; swelling properties

### ANNEX

#### (Mandatory Information)

#### A1. PROCEDURE TO CALIBRATE THE DENSITY OF SPHERICAL GLASS BEADS AND DETERMINE THE RADIUS OF DILATOMETER RETORT TUBES

##### A1.1 Scope

A1.1.1 This procedure provides a means to determine the average radius of a dilatometer retort tube.

##### A1.2 Summary of Procedure

A1.2.1 The average radius of a dilatometer retort tube is calculated from a measured volume of spherical glass beads which have a determined density.

##### A1.3 Significance and Use

A1.3.1 Coal expansion (dilatation) is a measure of the final volume occupied by the char of a coal pencil which was heated in a prescribed fashion. The average radius of the dilatometer retort tube is of great significance since char volume is equal to  $\pi \times$  measured chart height  $\times$  (retort tube radius).<sup>2</sup>

A1.3.2 This procedure enables the measurement of an average dilatometer retort tube radii. Rods of calibrated diameter are capable of measuring only cylinders that have a uniform diameter.

##### A1.4 Apparatus

A1.4.1 *Calibration Plugs*—shall be at least 20 mm in length and shall have a diameter of 7.99 and 8.01 mm  $\pm$  0.005 mm, respectively.

A1.4.2 *Calibration Retort Tube and Matching Piston*—shall consist of a seamless tube of steel, with an internal diameter of 8.00  $\pm$  0.005 mm and an external diameter of 14.50  $\pm$  0.05 mm. It shall be threaded to accept a gastight threaded plug at its base and shall have a minimum length of 345 mm.

A1.4.2.1 The piston shall be machined from steel rod to a diameter of 7.80  $\pm$  0.05 mm and extend at least 60 mm above the top of the retort tube when assembled with the matching calibrated retort tube. The piston shall weigh 100  $\pm$  10 g.

A1.4.2.2 The calibration retort tube and piston shall be marked and used as a matched set and require identification imprinted on both the retort tube and its matched piston. A line, called the Basement Level Reference Mark (BLRM), shall be made on the piston, such that when it is assembled with its matched calibration retort tube, with plug in place, the BLRM is even with the top of the retort tube. The length from the bottom of the piston to the BLRM is the length of retort tube bore with plug in place.

A1.4.2.3 This calibration retort tube and matching piston shall be used exclusively for determining the density of the glass spheres.

A1.4.3 *Balance*—shall have a weighing range of 100 g with 0.001-g readability.

A1.4.4 *Tapping Rod*—shall be made of mild steel and shall be 6.35 mm (0.25 in.) in diameter by 152.4 mm (6.0 in.) long.

### A1.5 Materials

A1.5.1 *Glass Spheres*—10 g of 1.00- to 1.40-mm-diameter (No. 14–18 mesh) sized glass spheres.

### A1.6 Determine the Density of the Glass Spheres

A1.6.1 Verify the diameter of the calibration retort tube by attempting to pass the 7.99- and 8.01-mm diameter-calibrated plugs through the bore of the retort tube. The calibration retort tube is acceptable if the 7.99-mm-diameter plug passes through the retort tube evenly and the 8.01-mm-diameter plug does not enter either end of the retort tube.

A1.6.2 Determine the density of the glass spheres. This portion of the procedure shall be performed in duplicate using an identified batch of glass spheres weighing no more than 100 g.

A1.6.2.1 Assemble the calibration retort tube and threaded base plug tightly together. Place the assembled calibration retort tube upright in a vise. Transfer approximately 10 g of glass spheres into the calibration retort tube.

A1.6.2.2 Place the matching piston into the calibration retort tube and record the height of the glass spheres using the BLRM as H1. Remove the assembly from the vise and tap the bottom 180 mm of the calibration retort tube at least 16 times with the tapping rod. Record the BLRM height as H2. Repeat the tapping as previously outlined and record the BLRM height as H3. The final BLRM height (H3) is to be recorded as  $H_{gs}$  provided that the threaded base plug is still tight and has not moved from its original position and  $H2 < H1$  and  $H3 = H2 \pm 0.5$  mm.

A1.6.2.3 Record the weight of an envelope as W1. Completely transfer the glass spheres into the envelope and record the weight as W2.

A1.6.2.4 Return the glass spheres into the identified batch and repeat this procedure.

### A1.7 Determine the Average Radius of the Dilatometer Retort Tube

A1.7.1 The dilatometer retort tube and its matching piston are used for dilatation analysis and shall be marked and used as a matched set as described in 6.1.3 of this test method.

A1.7.2 Assemble the retort tube and threaded base plug tightly together. Place the assembled retort tube upright in a vise. Transfer approximately 10 g of glass spheres into the retort tube.

A1.7.3 Place the matching piston into the retort tube and record the height of the glass spheres, using the BLRM, as Ht1. Remove the assembly from the vise and tap the bottom 180 mm of the retort tube at least 16 times with the tapping rod. Record the BLRM height as Ht2. Repeat the tapping as previously outlined and record the BLRM height as Ht3. The final BLRM height (Ht3) is to be recorded as  $Ht_{gs}$  provided that the threaded base plug is still tight and has not moved from its original position and  $Ht2 < Ht1$  and  $Ht3 = Ht2 \pm 0.5$  mm.

A1.7.4 Record the weight of an envelope as Wt1. Completely transfer the glass spheres into the envelope and record the weight as Wt2.

A1.7.5 Return the glass spheres into the identified batch and repeat this procedure.

### A1.8 Calculation

A1.8.1 *Calculation of the Density of the Glass Spheres:*

A1.8.1.1 Calculate the mass of the glass spheres,  $W_{gs}$ , as follows:

$$W_{gs} = W2 - W1 \quad (A1.1)$$

where:

$W_{gs}$  = the mass of glass spheres, g;

$W2$  = the mass of the envelope and the glass spheres, g, determined in A1.6.2.3; and

$W1$  = the mass of the envelope, g, determined in A1.6.2.3.

A1.8.1.2 Calculate the density of the glass spheres,  $D_{gs}$ , as follows:

$$D_{gs} = W_{gs}/(H_{gs} \times R_4^2) \quad (A1.2)$$

where:

$D_{gs}$  = the density of the glass spheres,  $g/mm^3$ ;

$W_{gs}$  = the mass of glass spheres, g, determined in A1.8.1.1;

$H_{gs}$  = the height occupied by the glass spheres in the calibration retort tube, mm, determined in A1.6.2.2; and

$R_4$  = the radius of the calibration retort tube = 4 mm.

A1.8.2 *Calculate the Average Radius of the Dilatometer Retort Tube:*

A1.8.2.1 Calculate the mass of the glass spheres,  $Wt_{gs}$ , as follows:

$$Wt_{gs} = Wt2 - Wt1 \quad (A1.3)$$

where:

$Wt_{gs}$  = the mass of glass spheres, g;

$Wt2$  = the mass of the envelope and the glass spheres, g, determined in A1.7.3; and

$Wt1$  = the mass of the envelope, g, determined in A1.7.3.

A1.8.2.2 Calculate the volume of the glass spheres,  $Vol_{gs}$ , as follows:

$$Vol_{gs} = Wt_{gs}/D_{gs} \quad (A1.4)$$

where:

$Vol_{gs}$  = the volume occupied by the glass spheres,  $mm^3$ ;

$Wt_{gs}$  = the mass of glass spheres as calculated in A1.8.2.1; and

$D_{gs}$  = the density of the glass spheres as calculated in A1.8.1.2.

A1.8.2.3 Calculate the average radius of the dilatometer retort tube,  $R_{av}$ , as follows:

$$R_{av} = -\sqrt{[Vol_{gs}/(\pi \times Ht_{gs})]} \quad (A1.5)$$

where:

$R_{av}$  = the average radius of the dilatometer retort tube, mm;

$Vol_{gs}$  = the volume occupied by the glass spheres,  $mm^3$ , as calculated in A1.8.2.2; and

$Ht_{gs}$  = the measured height of the glass spheres, mm, as determined in A1.7.2.

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