**Designation: ~~D5515 – 21~~ D5515 – 22**

# Standard Test Method for Determination of the Swelling Properties of Bituminous Coal Using a Dilatometer[1]

This standard is issued under the fixed designation D5515; 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.

## 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 mass 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 ≥1 as determined in accordance with Test Method D720.

1.3 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

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

1.5 *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.*

[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.

Current edition approved ~~April 1, 2021~~Sept. 1, 2022. Published ~~April 2021~~September 2022. Originally approved in 1994. Last previous edition approved in ~~2020~~2021 as ~~D5515 – 20.~~D5515 – 21. DOI: ~~10.1520/D5515-21.~~10.1520/D5515-22.

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]
D720 Test Method for Free-Swelling Index of Coal
D2013 Practice for Preparing Coal Samples for Analysis
D2234/D2234M Practice for Collection of a Gross Sample of Coal

2.2 *ISO Standards:*[3]
ISO 349 Hard Coal-Audibert-Arnu Dilatometer Test
ISO 8264 Hard Coal—Determination of the Swelling Properties Using a Dilatometer

## 3. Terminology

3.1 *Definitions:*

3.1.1 *air-dried coal pencil mass, $M_{dry}$ ~~—~~, n—*the calculated mass of the trimmed 60 mm long coal pencil corrected for added water, expressed in grams.

3.1.2 *basement level reference mark height, $BLRM_{ht2}$ ~~—~~n—*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.

3.1.3 *coal ~~pencil—~~pencil, n—*a 60 mm long test specimen formed by compression in a mold from coal which has been pulverized to pass a 250 µm (No. 60 U.S. standard sieve) sieve.

3.1.4 *equivalent percent dilatation for 2.50 g of air dried coal, $\%D_{2.50}$ ~~—~~n—*the calculated percent expansion for a 2.50 g, unmoistened, 60 mm long, coal pencil corrected for average tube radii, expressed as a percentage.

3.1.5 *maximum contraction temperature, ~~T2—~~T2, n—*the temperature at which the coal pencil starts swelling, expressed in degrees Celsius (see Fig. 1*a* and *b*).

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

3.1.6 *maximum dilatation temperature, ~~T3—~~T3, n—*the temperature at which the coal pencil first reaches a maximum height after swelling, that does not change by more than 1 *%D* during the next 3 °C, expressed in degrees Celsius (see Fig. 1*a* and *b*).

3.1.6.1 *Discussion—*
For coals that exhibit no dilatation, *T3* should be reported as *contraction only* (see Fig. 1*c* and *d*). For coals that the maximum dilatation exceeds 300 %, *T3* should be reported as the temperature at which the recorded height reaches 300 % and as *taken at 300 %D*.

3.1.7 *percent contraction, ~~%C—~~%C, n—*the minimum recorded height of the char expressed as a positive whole number percentage, based on an initial coal pencil height of 60 mm (see Fig. 1*a* through *c*).

3.1.7.1 *Discussion—*
For coals that exhibit expansion following contraction, the percent contraction is the last minimum recorded height. For coals that exhibit contraction only and are still contracting at 500 °C, *%C* is the height recorded at 500 °C (see Fig. 1*d*).

3.1.8 *percent dilatation, ~~%D—~~%D, n—*the maximum recorded height of the char taken at the maximum dilatation temperature, *T3*, expressed as a whole number percentage, based on an initial coal pencil height of 60 mm.

3.1.8.1 *Discussion—*
For coals which the maximum recorded height is above the initial 60 mm baseline, *%D* is expressed as a positive percentage (see Fig. 1*a*). For coals which the maximum recorded height is below the initial 60 mm baseline, *%D* is expressed as negative percentage (see Fig. 1*b*). For coals that exhibit no dilatation, *%D* should be reported as *contraction only* (see Fig. 1*c* and *d*). For percent dilatation measurements exceeding 300 %, report *%D* as *greater than (>) 300 %*.

[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.
[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

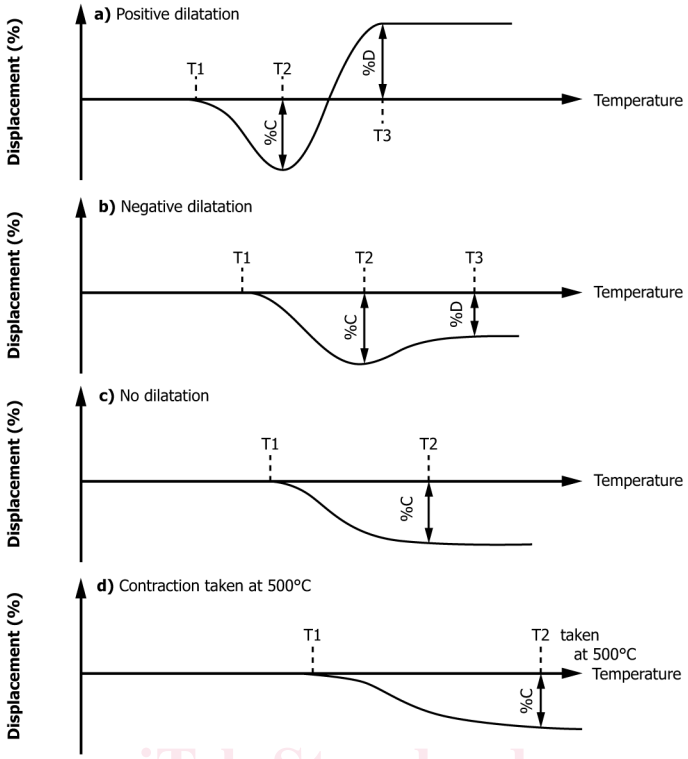



**FIG. 1 Types of Dilatation Curves**



3.1.9 *percent water added, $\%_{H_2O}$*~~=~~, *n*~~—~~the mass fraction of water added to the coal during making of the pencil, expressed as a percent.

3.1.10 *softening temperature, ~~T1—~~T1, n*~~—~~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 degrees Celsius (see Fig. 1).



3.1.11 *temperature ~~range—~~range, n*~~—~~difference between the maximum dilatation temperature, *T3*, and the softening temperature, *T1*.

3.1.11.1 *Discussion*~~—~~

For coals that exhibit no dilatation, and therefore *T3* is not recorded, the temperature range shall be reported as *range not available*. For coals where the maximum dilatation exceeds 300 %, the temperature range shall be reported as *greater than (>)* the value of the difference of *T3* taken at 300 %D and the softening temperature, *T1*.

3.1.12 *wet coal pencil mass, $M_{wet}$*~~=~~*n*~~—~~the measured mass of a trimmed 60 mm long coal pencil mass, expressed in grams.

## 4. Summary of Test Method

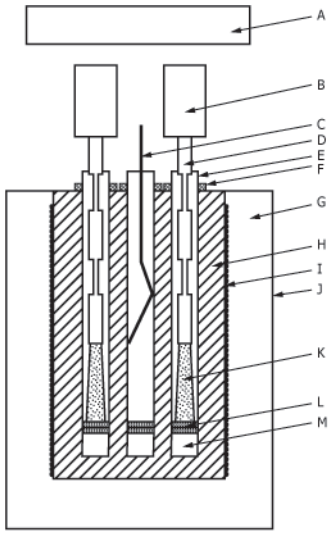
4.1 The test involves preparing a coal pencil and determining the changes of the coal pencil height in a retort tube during a prescribed heating cycle.

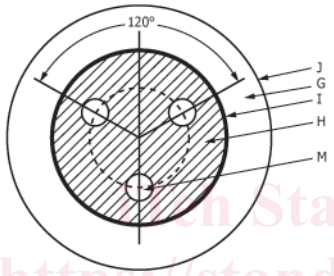
## 5. Significance and Use

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:




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 mm ± 0.1 mm diameter on hole center radius of 20.0 mm)

**FIG. 2 Typical Dilatometer Apparatus**



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 mm 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 °C ± 0.1 °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 mm ± 0.05 mm and an external diameter of 14.50 mm ± 0.05 mm. It shall have a support collar at the top and be threaded to accept a gas-tight, threaded plug at its base. The retort tube shall have a minimum 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 mm ± 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 g ± 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 mm ± 0.5 mm long coal pencil. The

mold shall have a 70.0 mm ± 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 mm ± 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 a 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~~ capacity of 160 g with 0.01 g readability.

## 7. Preparation of Sample

7.1 Collect a gross sample in accordance with the requirements of Test Method D2234/D2234M.

7.2 The analysis sample shall consist of a minimum of 50 g of coal pulverized to 250 µm (No. 60 U.S. standard sieve) sieve in accordance with Test Method D2013. Perform the dilatation analysis on the coal sample passing 250 µm (No. 60 U.S. standard sieve) sieve within five days after reduction to 250 µm (No. 60 U.S. standard sieve) 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 $BLRM_{ht}$ 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):

8.2.1 Temperatures measured in Bores 1 and 2 must be ±3 °C measured at the same height for 0 mm, 45 mm, 90 mm, 135 mm, and 180 mm above the retort tube plug.



8.2.2 Temperatures measured between the control thermocouple placed at 60 mm above the retort tube plug and those of Bores 1 and 2 shall be ±3 °C measured at 0 mm, 45 mm, 90 mm, and 135 mm and ±6 °C at 180 mm.

8.3 The dilatometer temperature readout of the measuring thermocouple shall be checked 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 The furnace temperature profile and measuring thermocouple calibration shall be verified at a minimum interval of every 400 determinations or 6 months, whichever occurs first, or upon any furnace component being repaired or replaced.

8.5 Calibrate the retort tubes, as described in Annex A1, prior to initial use and after every 50 tests.

## 9. Preparation of the Coal Pencil

9.1 All masses 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 interruption.

9.1.1 *Preparation of Homogeneous Coal Sample and Water Mix*—The fraction of water in the mix shall be ≤11 % on a mass fraction 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 pre-weighed 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. If a break in the pencil occurs at any point during its construction, the pencil shall be remade. 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.

10.4 Attach the mechanism used to measure piston height and adjust the piston height reading to 0 *%D* ± 5 *%D*.

10.5 The heating control shall be set so that the furnace temperature recovers to the 315 °C setpoint within 10 min of loading the retort tubes and piston assemblies. Start the temperature control program immediately once the setpoint temperature is reached.

10.6 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 °C ± 5 °C. Remove the piston height-measuring mechanism. Remove the retort tube and piston assemblies as a unit. Measure and record the $BLRM_{ht}$ for each piston/retort tube assembly. Allow the assembly to cool to room temperature before cleaning.

10.7 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, $\%_{H_2O}$, as follows:

$$\%_{H_2O} = [100 \times (M3 - M2)] / [(M2 - M1) + (M4 - M3)] \quad (1)$$

where:

~~100 = conversion factor from dimensionless mass fraction to percent, %;~~
100 = conversion factor from dimensionless mass fraction to percent, %,
~~$\%_{H_2O}$ = mass fraction of water added to the coal, %;~~
$\%_{H_2O}$ = mass fraction of water added to the coal, %,
~~*M3* = mass of the mixing vessel plus first coal increment plus water, g;~~
*M3* = mass of the mixing vessel plus first coal increment plus water, g,
~~*M2* = mass of the mixing vessel plus first coal increment, g;~~
*M2* = mass of the mixing vessel plus first coal increment, g,
~~*M1* = mass of the mixing vessel, g; and~~
*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, $M_{wet}$, as follows:

$$M_{wet} = (M5 - M6) \quad (2)$$

where:

~~$M_{wet}$ = mass of the prepared coal pencil, g;~~
$M_{wet}$ = mass of the prepared coal pencil, g,
~~*M5* = mass of the trimmed coal pencil and trimming block, g; and~~
*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, $M_{dry}$, as follows:

$$M_{dry} = M_{wet} - (M_{wet} \times \%_{H_2O} \times 0.01) \quad (3)$$



where:

~~$M_{dry}$ = mass of air-dried coal in the coal pencil, g;~~
$M_{dry}$ = mass of air-dried coal in the coal pencil, g,
~~$M_{wet}$ = mass of the prepared coal pencil calculated in 12.2, g;~~
$M_{wet}$ = mass of the prepared coal pencil calculated in 12.2, g,
~~$\%_{H_2O}$ = mass fraction of water added to the coal calculated in 12.1, %; and~~
$\%_{H_2O}$ = mass fraction of water added to the coal calculated in 12.1, %, and
0.01 = conversion factor from percent, %, to dimensionless mass fraction.

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

$$H_c = \%D \times 0.6 + 60 \quad (4)$$

where:

~~$H_c$ = calculated char height, mm;~~
$H_c$ = calculated char height, mm,
~~*%D* = dilatation for a 60 mm long trimmed coal pencil, %;~~
*%D* = dilatation for a 60 mm long trimmed coal pencil, %,
~~0.6 = the factor used to convert *%D* to a height, mm/%; and~~
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:

$$H_{2.50} = (H_c / M_{dry}) \times 2.50 \quad (5)$$