

Designation: D 167 – 93 (Reapproved 1999)<sup>€1</sup>

# Standard Test Method for Apparent and True Specific Gravity and Porosity of Lump Coke<sup>1</sup>

This standard is issued under the fixed designation D 167; 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.

 $\epsilon^1$  Note—Sections 15, 16.1.1, and 16.1.2 were editorially changed in September 1999.

# 1. Scope

1.1 This test method describes the determination of apparent specific gravity (Sections 2 to 9) and true specific gravity (Sections 10 to 13) of lump coke larger than 25-mm (1-in.) size and calculating porosity (Section 14) from the specific gravity data.

1.2 The values given in SI units shall be regarded as the standard. Inch-pound units shall be accepted on an equivalent basis.

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.

## APPARENT SPECIFIC GRAVITY

#### 2. Referenced Documents

- 2.1 ASTM Standards:
- D 121 Terminology of Coal and Coke<sup>2</sup>
- D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis<sup>2</sup>
- D 4621 Guide for Accountability and Quality Control in the Coal Analysis Laboratory<sup>2</sup>
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes<sup>3</sup>
- E 323 Specification for Perforated-Plate Sieves for Testing Purposes<sup>3</sup>

# 3. Significance and Use

3.1 Apparent and true specific gravity, as determined by this test method, are influenced by the type of coals carbonized and the operating and preparational conditions of that carboniza-

tion, that is, charge bulk density, heating rate, and pulverization level. In turn, these properties directly influence the performance in processes using coke.

## 4. Apparatus

4.1 The apparatus for the determination of the apparent specific gravity of coke shall consist of the following:

4.1.1 An Elliptical or Rectangular Cross-Sectioned Container, approximately 560 mm (22 in.) in length, 280 mm (11 in.) in width, and a minimum of 330 mm (13 in.) in height, provided with a spout consisting of a short 13-mm ( $\frac{1}{2}$ -in.) nipple extending horizontally from the container about 270 mm ( $10\frac{1}{2}$  in.) above the bottom.

4.1.2 *Wire Cage or Basket*, of about 13-mm (<sup>1</sup>/<sub>2</sub>-in.) squaremesh screen wire cloth provided with a cover and two long handles, suitable for holding the entire sample of coke and so made as to fit inside the container below the spout.

4.1.3 Bucket or Other Vessel, 11-L (3-gal), suitable for receiving the displaced water.

4.1.4 *Pan*, about 380 mm (15 in.) square and 76 mm (3 in.) in height or the equivalent for containing the coke during the determination of its weight.

4.1.5 Balance, sensitive to 0.05 kg (0.1 lb).

#### 5. Sampling at Source

5.1 When the porosity test is desired on run-of-oven coke, the sample shall be collected from the coke wharf.

5.1.1 *By-Product Coke*—About 23 kg (50 lbs) of representative pieces of coke shall be selected from the coke wharf for each test. This is best accomplished by dividing the coke on the wharf into approximately equal areas and selecting an equal number of pieces from each area. Each piece of coke selected shall be approximately equal in length to one half of the width of the coke ovens, and shall show a "cauliflower" end produced at the walls of the ovens, and an "inner" end produced at the center of the ovens.

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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 05.06.

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

# 6. Sampling at Delivery

6.1 If the porosity test is desired on coke for furnace or cupola use, the sample shall be representative of the material in question and collected at the place of delivery.

6.1.1 *By-Product*—The sample is best collected as the coke is delivered from the railroad cars into the bins. This can be accomplished by securing a representative sample of 4.5- to 6.8-kg (10- to 15-lbs) capacity in the coke stream at regular intervals during the period of unloading. The sample collected shall be large enough to give about 23 kg (50 lbs) of coke pieces, none of which would in any position pass through a 25-mm (1-in.) square-mesh sieve.

Note 1—Since the cage or basket is of 13-mm ( $\frac{1}{2}$ -in.) square-mesh screen wire cloth, it is necessary to have pieces that will remain in the cage when it is removed from the water.

6.1.2 Since it is very difficult to collect a representative sample from coke exposed in bins and cars, care shall be taken to take pieces representing the entire exposed area, if sampling must be done in this manner. It is best accomplished by dividing the exposed surface to be sampled into approximately equal areas, and selecting an equal number of pieces from each area. A 23-kg (50-lbs) sample of representative pieces shall be collected, none of which shall in any position pass through a 25-mm (1-in.) square-mesh sieve.

# 7. Calibration

7.1 Calibrate the Hogarth specific gravity bottle to determine the mass of the bottle and the distilled water required to fill it (variable P). This can be done by (I) constructing a table graph of actual mass measurements of the bottle filled with distilled water at various temperatures or (2) using the data in the tables of corrections for determining the true capacities of glass vessels from the mass of water in air (Lange or CRC Handbooks).

## 8. Procedure

8.1 Select about 11.4 kg (25 lbs) of coke from the sample so that it is representative of the material under consideration with regard to size, shape, and general appearance. Dry the coke to constant mass at a temperature of from 105 to  $200^{\circ}$ C. Weigh the coke when cool, after shaking and brushing off any adhering dust.

8.2 Place a cork in the spout of the container, which has been placed on a level and rigid base or floor. After the empty cage has been placed into the container, pour water at room temperature into the container until the water level is above the spout. After the water has come to rest, remove the cork from the spout and permit the excess water to drain out for 1 min after the overflow stream starts to discharge drop by drop. Then replace the cork and remove the cage from the water, care being taken to shake all adhering water back into the container. Then place the weighed dried coke sample into the cage and after fastening the cover, lower the cage containing the coke into the water.

NOTE 2—If there is not sufficient capacity in the container above the spout to retain the displaced water, some of the water may be drawn off into a weighed bucket, or other suitable vessel, by removing the cork from the spout while the coke is being lowered.

8.3 Permit the cage to remain in the water for 15 min, with occasional shaking to detach any air bubbles adhering to the surface of the coke, care being taken not to disturb the position of the container. At the end of the 15-min period, during which the coke shall have been completely submerged at all times, remove the cork after the water has come to rest and permit the displaced water to drain into a weighed bucket or other suitable vessel for 1 min after the overflow stream starts to discharge drop by drop. Replace the cork, remove the cage containing the coke from the water and permit it to drain for 1 min. Remove the wet coke from the cage and weigh it.

8.4 Determine the mass of the displaced water, which has been caught in the bucket.

## 9. Calculation

9.1 Calculate the apparent specific gravity as follows:

Apparent specific gravity = A/[B + (C - A)]

where:

A = mass of dry coke,

B = mass of water displaced by wet coke, and

C = mass of wet coke.

# TRUE SPECIFIC GRAVITY

#### 10. Apparatus

10.1 The apparatus for the determination of the true specific gravity of coke passing a 75- $\mu$ m (No. 200) sieve shall consist of a Hogarth's specific gravity bottle with side tubulure, having a capacity of about 100 mL.

#### **11. Sample Preparation**

11.1 Select about 4.5 kg (10 lbs) of coke from the sample (see Section 5) so that it is representative of the material under consideration with regard to size, shape, and general appearance. Crush the coke to pass through a 6.3-mm ( $\frac{1}{4}$ -in.) screen and split the sample size to about 2.3 kg (5 lbs). Crush this fraction to pass through a 850-µm (No. 20) sieve and further split the sample to approximately 200 g. Grind the 200-g fraction to pass through a 250-µm (No. 60) sieve and split the sample size to approximately 50 g. Pulverize this entire fraction to minus 75-µm (No. 200) and dry for 1 h at 105°C.

NOTE 3—It may be necessary to dry the sample before crushing or pulverizing during the sample preparation.

NOTE 4—Equipment used for reducing the sample particle size and quantity is described in Method D 346. In all cases, care must be taken to select equipment that will not abrade and add unwanted impurities to the sample during the size-reduction steps.

## **12. Procedure**

12.1 Carefully introduce a 10-g portion of 75- $\mu$ m (200mesh) coke, which has been previously dried for 1 h at 105°C, into the specific gravity bottle with a sufficient quantity of distilled water to fill the bottle about one half full. Bring to a boil, keeping the contents boiling for 1 h. Shake the specific gravity bottle frequently so as to wash down any coke adhering to the sides. Remove the bottle from the plate, after boiling for 1 h, fill it to the tubulure with recently boiled and cooled distilled water, and insert the stopper. Permit the bottle to stand until the contents have cooled to room temperature (Note 5); then fill the bottle to slightly above the mark on the capillary of the stopper with recently boiled distilled water which has been cooled to room temperature (Note 6). Adjust the water level to the mark on the capillary by touching a piece of filter paper to the end of the tubulure. Then wipe the bottle dry and weigh it immediately. Immediately after the weighing, remove the stopper and take the temperature of the contents. This temperature is used to determine the exact capacity of the Hogarth bottle (10.1).

Note 5-Cooling may be hastened by placing the bottle in water.

NOTE 6—This is conveniently done by inserting the end of the tubulure in a small beaker of the distilled water and applying a slight suction on the stopper.

## 13. Calculation

13.1 Calculate the true specific gravity as follows:

True specific gravity = 
$$W/[W - (W' - P)]$$

where:

W = mass of dry coke,

- W' = mass of the bottle and the dry coke and water required to fill it, and
- P = mass of the bottle and the dry coke and water required to fill it.

## POROSITY

## 14. Calculation

14.1 Calculate the porosity (percentage of cell space) of lump coke from the apparent specific gravity of the moisture-

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free lump coke and the true specific gravity of the moisturefree coke passing a 75-µm (No. 200) size, as follows:

$$Porosity = 100 - 100 \text{ (apparent sp. gr./true sp. gr.)}$$
(1)

## 15. Report

15.1 Apparent specific gravity and true specific gravity shall be reported to the nearest 0.01. Porosity is reported to the nearest whole percent.

# 16. Precision and Bias

16.1 Both Specific Gravity Methods:

16.1.1 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than 0.01.

16.1.2 *Reproducibility*—Results submitted by each of two laboratories should be considered suspect if they differ by more than 0.01.

16.1.3 *Bias*—Since these test methods are empirical standards, the degree of their absolute bias cannot be determined.

## 17. Keywords

17.1 apparent specific gravity; coke; porosity; true specific gravity