Designation: D5004 – 89 (Reapproved 2004)ε1

Standard Test Method for Real Density of Calcined Petroleum Coke by Xylene Displacement[1]

This standard is issued under the fixed designation D5004; 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.

ε[1] NOTE—Warning notes were moved into text editorially in November 2004.

1. Scope

1.1 This test method is intended for the determination of the real density (RD) of calcined petroleum coke. Real density, by definition, is obtained when the particle size of the test specimen is smaller than 75 µm (No. 200 sieve).

1.2 The values stated in SI units are to be regarded as 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.* For specific warning statements, see Sections 10 and 11.1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

D1193 Specification for Reagent Water

D2013 Practice for Preparing Coal Samples for Analysis

D2234/D2234M Practice for Collection of a Gross Sample of Coal

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4292 Test Method for Determination of Vibrated Bulk Density of Calcined Petroleum Coke

D4930 Test Method for Dust Control Material on Calcined Petroleum Coke

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 *Definitions:*

3.1.1 *calcined coke*—green petroleum coke that has been thermally treated to drive off the volatile matter and to develop crystalline structure.

3.1.2 *petroleum coke*—a solid, carbonaceous residue produced by thermal decomposition of heavy petroleum fractions or cracked stocks, or both.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *bulk density*—the mass of the particles divided by the volume they occupy that includes the space between the particles. Refer to Test Method D4292 for bulk density procedures.

3.2.2 *dedusting material*—see Test Method D4930.

3.2.3 *real density*—(also referred to as true specific gravity), the mass divided by the volume occupied by the material excluding pores and voids. It is required, therefore, that voids in the coke be eliminated and that pores in the material be filled by the fluid being displaced. This requirement is met for the purposes of this test method by reducing the coke particles to a size smaller than 75 µm.

3.2.3.1 *Discussion*—The density of particles larger than 75 µm up to the largest that can be put into the helium pycnometer can also be determined, but must be designated as particle density (PD). The precision data obtained for RD may not be applicable to PD.

4. Summary of Test Method

4.1 The mass of the sample is determined directly and the volume derived by determining the mass of liquid displaced when the sample is introduced into a pycnometer.

$$RD = M \times D/L \quad (1)$$

where:

M = mass of sample,

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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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.

D = density of displaced liquid, and
L = mass of displaced liquid.

5. Significance and Use

5.1 The density of petroleum coke directly influences the physical and chemical properties of the manufactured carbon and graphite artifacts for which it is used. Density, therefore, is a major quality specification of calcined petroleum coke and is used as a control in coke calcination.

6. Interferences

6.1 Oil or other material sprayed on calcined petroleum coke to control dust will interfere with the determination of real density so the oil must be removed before reducing the sample to 75 µm.

6.1.1 When a petroleum oil was used, it can be removed by flushing with a solvent such as methylene chloride, dichloroethane, or toluene. The solvent must be completely removed before proceeding with the RD determination. Heating to 10°C above the boiling point of the solvent used or application of vacuum is satisfactory for the removal of the dedusting oil.

NOTE 1—Consult the Material Safety Data Sheet (MSDS) for the selected solvent.

6.1.2 An alternative method of oil removal is by heating the calcined coke sample in an oven at 700°C for 1 h.

7. Apparatus

7.1 *Pycnometer*, or specific gravity bottle, 50 mL, with a ground glass stopper with a capillary hole.[3] Bottles with a large neck (12 to 13 mm outside diameter) are preferred.

7.2 *Water Bath*, controlled to a temperature of 25 ± 0.1°C.

NOTE 2—This test method is written to be performed at 25 ± 0.1°C; however, some laboratories may not have the provisions to perform the test at this temperature. It is permissible to perform the test procedure at any temperature between 20 and 40°C providing that the water bath is controlled at ±0.1°C of the chosen temperature and the pycnometers are calibrated at the same temperature that is used to determine the real density of the petroleum coke sample. This is possible due to the fact that the real density of calcined petroleum coke is not affected by temperature changes over a limited temperature range.

7.3 *Analytical Balance*, accurate to ±0.1 mg.

7.4 *Vacuum Desiccator*, with guard, connected to a vacuum source capable of lowering pressure to 75 mm of Hg (10 kPa).

7.5 *Desiccator*, with drying agent. Anhydrous calcium sulphate is satisfactory.

7.6 *Drying Oven*, preferably a vacuum oven, for temperature to 120°C.

7.7 *Lead Weights*, for the pycnometers, to prevent tipping over in the water bath. These can be made by coiling solid wire solder.

7.8 *Wire Sieve*, 75 µm (No. 200 mesh), meeting Specification E11.

8. Reagents

8.1 *Purity of Water*—References to distilled water shall be understood to mean reagent water as defined by Type III of Specification D1193.

8.2 Analytical reagent grade solvents are not required but can be used. The technical grade of each of the following is satisfactory:

8.2.1 *Acetone*, *Xylene*, and *Ethyl Alcohol.* (See 6.1.2.)

9. Sample Preparation

9.1 For recommended practice for obtaining, handling, and preparing coke samples, refer to Practice D346, Method D2013, Test Methods D2234/D2234M, and Practice D4057. See Section 6.

9.2 Crush 50 g of coke so that the entire sample will pass through a 75 µm (No. 200) sieve. Dry the crushed sample in a drying oven at 115± 5°C to constant mass (approximately 8 h). Cool in a desiccator.

NOTE 3—Constant mass is considered to be achieved when change in mass is less than ±0.05 g after a 30 min test drying period.

10. Pycnometer Calibration (Determination of Pycnometer Volume)

10.1 Clean the pycnometer and its stopper with detergent, rinse thoroughly with water then with acetone. Place in a desiccator to dry, then weigh the empty pycnometer together with its stopper to 0.1 mg (mass W_o). The temperature of the pycnometer is to be close to room temperature when its weight is determined. (**Warning—**Commercial pycnometers (specific gravity bottles) can either have not been calibrated at 25°C or else not calibrated to the accuracy required for this test method, so it is necessary that the pycnometer volume be determined.)

NOTE 4—Do not handle the pycnometer with bare fingers. Finger cots or surgical gloves can be worn, or tongs can be used, when handling the pycnometer to prevent moisture from fingers influencing the weight.

10.2 Fill the pycnometer with freshly boiled (to remove air) and cooled distilled water, and replace the stopper. Immerse the pycnometer up to the neck in the 25° ± 0.1°C water bath for 1 h. Use the lead weights to prevent tipping. Replace water that leaves the capillary during this period. A syringe is convenient for this purpose.

10.3 At the end of the temperature stabilization period, check the capillary to be certain it is completely filled. Remove excess water on the stopper by dabbing with filter paper. If water is inadvertently removed from the capillary it must be replaced. Remove the pycnometer from the 25°C bath, rinse immediately with acetone, dry, and weigh to 0.1 mg (mass W_3).

NOTE 5—Avoid any heating after the pycnometer is removed from the 25°C bath. Heating will expand the water and cause loss from the capillary. Water is not to be added to the capillary after the pycnometer is removed from the 25°C bath. The purpose for the immediate acetone rinse is to contract the contents so it will recede in the capillary. Ethyl alcohol can be used in place of acetone. If laboratory temperature is 25°C or above, a water bath maintained at about 20°C should be provided to cool the pycnometer for about 1 min then dry and weigh. Do not chill the pycnometers so cold that moisture from the atmosphere will condense on them and make accurate weighing impossible.

10.4 Calculate the volume, *v*, of the pycnometer in cubic centimetres using Eq 2. Round off to 0.001 cm^3.

$$v = \frac{W_3 - W_o}{\rho w} \quad (2)$$

[3] A Gay-Lussac pycnometer has been found suitable for this purpose.