Designation: D2638 – 10

Standard Test Method for Real Density of Calcined Petroleum Coke by Helium Pycnometer[1]

This standard is issued under the fixed designation D2638; 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 the determination of the real density (RD) of calcined petroleum coke. Real density, by definition is obtained when the particle size of the specimen is smaller than 75 microns (No. 200 Sieve).

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 *calcined petroleum coke*, *n*—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 which 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*—(RD) (also be 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 microns.

3.2.3.1 *Discussion*—The density of particles larger than 75 microns 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 A representative sample of calcined petroleum coke is dried and ground to pass a 75-micron (200-mesh) screen. The mass of the sample is determined directly and the volume derived by the volume of helium displaced when the sample is introduced into a helium pycnometer. The ratio of the mass of the sample to the volume is reported as the real density.

5. Significance and Use

5.1 The real density of calcined 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.

[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.

Current edition approved May 1, 2010. Published July 2010. Originally approved in 1991. Last previous edition approved in 2006 as D2638–06. DOI: 10.1520/D2638-10.

[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.

***A Summary of Changes section appears at the end of this standard.**

6. Interferences

6.1 Oil or other dedust 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 microns. Refer to Test Method D4930 for dedust oil removal.

7. Apparatus

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

7.2 *Desiccator*.

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

7.4 *Helium Pycnometer*:

7.5 *Jaw Crusher and Roll Crusher*—Other style crushers which allow control over particle size without contamination are acceptable.

7.6 *Rifflers*, with hoppers and closures.

7.7 *Wire Sieve*, 75 microns (No. 200 mesh), meeting Specification E11.

8. Reagents and Materials

8.1 Helium, 99.9 %.

9. Sample Preparation

9.1 For recommended practice for obtaining, handling, and preparing coke samples, refer to Test Methods D346, D2013, D2234/D2234M, D4057, and D4930. The equipment and procedures for crushing and dividing are also described in these test methods.

9.2 Reduce and divide the gross sample to obtain a laboratory analysis sample.

9.3 Divide, by riffling, a minimum of 100 g from the laboratory analysis sample.

NOTE 1—The amount of coke required will be dictated by the operating instructions supplied by the helium pycnometer manufacturer. However, to assure that the test sample is representative of the entire sample, a minimum sample size of 100 grams is required.

9.4 When a dedusting oil is present it must be removed prior to further sizing. Refer to Test Method D4930 for dedust oil removal.

9.5 Crush 100 g of the test sample so that the entire sample will pass through a 75-micron (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.

10. Procedure

10.1 Place 5 to 150 g (80 to 90 % of the maximum capacity of the particular holder being used) of the prepared crushed, sized and dried test sample into the specimen holder provided with the instrument. Weigh to the nearest milligram.

10.2 Place the pre-weighed test sample and specimen holder into the sample chamber and evacuate.

10.3 Connect, either manually or automatically, the control chamber, containing helium at a higher pressure than the sample chamber, with the sample chamber. Determine the final equilibrated pressure.

10.4 The volume of the calcined petroleum coke sample is calculated from the known volume of the two chambers, the absolute pressures in each chamber prior to connection and the final pressure after the chambers have been connected.

10.5 Record the final sample volume or density when the pycnometer has reached equilibrium.

NOTE 2—This is a generic procedure which describes the basic steps involved to obtain a final result. For further specific details, the manufacturers' operating instructions[3] are to be reviewed.

11. Calculation

11.1 Determine the real density of the sample in g/cm^3 from Eq 1.

$$\text{Density} = M/V \quad (1)$$

where:

M = mass of sample in g, and

V = volume of helium displaced, cm^3.

12. Report

12.1 Report to the third decimal place the real density as calculated in 11.1.

13. Precision and Bias [4]

13.1 The values in the statements were determined in a cooperative program following Practice E691. In this 2008 study, there were twelve laboratories and eight samples. The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows:

13.1.1 *Repeatability*—The difference between successive results by the same operator using the same apparatus under constant operating conditions on the identical test materials, will in the long run, in normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$\text{Repeatability} = 0.005 \text{ g/cm}^3 \quad (2)$$

13.1.2 *Reproducibility*—The difference between two single and independent results obtained by different laboratories on identical test materials will in the long run, in normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$\text{Reproducibility} = 0.013 \text{ g/cm}^3 \quad (3)$$

13.1.3 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method for measuring the real density of calcined petroleum coke, no statement on bias is being made.

NOTE 3—The reproducibility value reflects the overall reproducibility covering all makes of pycnometers used in this work.

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[3] Specific manufacturer's instruction manuals and year of issue used in developing this test method are given in the following list.

Quantachrome: Model MVP-1, "Quantachrome Multipycnometer Instruction Manual," March 1987; Model SPY-2, "Quantachrome Stereopycnometer Instruction Manual," March 1982.

Beckman, Model 930, "Beckman Instructions," 1971.

Frank Jones, Model 204, "Coberly-Stevens Porosimeter Instructions," 1986.

Micromeritics: Model 1320, "Instruction Manual Autopycnometer 1320," March 1984, May 1984, February 1985; Model 1305, "Instruction Manual Multivolume Pycnometer 1305," May 1985.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1687.

14. Keywords

14.1 calcined petroleum coke; helium pycnometer; real density

SUMMARY OF CHANGES

Subcommittee D02.05 has identified the location of selected changes to this standard since the last issue (D2638–06) that may impact the use of this standard.

(*1*) Revised Section 13.

(*2*) Added Research Report and Footnote 4.