



Designation: ~~D2638 – 10 (Reapproved 2015)~~^{ε1} D2638 – 21

Standard Test Method for Real Density of Calcined Petroleum Coke by Helium Pycnometer¹

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.

^{ε1} NOTE — Editorial changes were made throughout in December 2015.

1. ~~Scope~~ 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 μm (U.S. 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~~ safety, health, and ~~health~~ environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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.*

2. Referenced Documents

[ASTM D2638-21](#)

<https://standards.iteh.ai/catalog/standards/sist/93ed24f4-1999-4497-8275-043c3ca18229/astm-d2638-21>

2.1 *ASTM Standards:*²

~~D346D4930 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis~~ [Test Method for Dust Control Material on Calcined Petroleum Coke](#)

~~D2013D6969 Practice for Preparing Coal Preparation of Calcined Petroleum Coke Samples for Analysis~~

~~D2234/D2234MD6970 Practice for Collection of a Gross Sample of Coal~~ [Calcined Petroleum Coke Samples for Analysis](#)

~~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:*

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

Current edition approved Oct. 1, 2015; Jan. 1, 2021. Published December 2015; January 2021. Originally approved in 1991. Last previous edition approved in 2010 as ~~D2638 – 10~~ D2638 – 10 (2015)^{ε1}. DOI: ~~10.1520/D2638-10R15E01~~ 10.1520/D2638-21.

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

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, n*—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, n*—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.

3.2.1 *dedusting material, n*—See Test Method [D4930](#).

3.2.2 *real density, n*—(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 μm .

3.2.2.1 *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 A representative sample of calcined petroleum coke is dried and ground to pass a 75 μm (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.

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 μm . 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 $^{\circ}\text{C}$.

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 *Riffles*, with hoppers and closures.

7.7 *Wire Sieve*, 75 μm (U.S. 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 Practices D346D6969, D2013, D2234/D2234M, D4057, and D4930D6970.~~ The equipment and procedures for crushing and dividing are also described in these ~~test methods/practices.~~

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

9.3 Divide, by riffing, 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 g 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 μm (No. 200) sieve. Dry the crushed sample in a drying oven at $115\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ to constant mass (approximately 8 h). Cool in a desiccator.

10. Procedure

10.1 Place 5 g 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 instructions³ 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

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.