Designation: D8097 – 23

# Standard Test Method for Determination of Bulk Density for Specific Size Fractions of Calcined Petroleum Coke Using a Transaxial Pressure Pycnometer[1]

This standard is issued under the fixed designation D8097; 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 The test method covers the determination of bulk density for a specific size fraction of calcined petroleum coke using an automated pycnometer that compacts coke by applying transaxial pressure under a controlled force.

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, health, and 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

2.1 *ASTM Standards:*[2]

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D6969 Practice for Preparation of Calcined Petroleum Coke Samples for Analysis

D6970 Practice for Collection of Calcined Petroleum Coke Samples for Analysis

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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

## 3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.1.2 *bulk density, n—of coke*, the ratio of the mass of a collection of particles of a specified particle size range to the volume occupied.

3.1.3 *transaxial pressure, n*—pressure applied across a horizontal axis.

## 4. Summary of Test Method

4.1 A representative sample of calcined petroleum coke is collected and then screened using Specification E11 sieves (8 mm, 4 mm, 2 mm, 1 mm, 0.5 mm, 0.25 mm) into fractions. Each specific size fraction is then weighed and the bulk volume is measured using the pycnometer. This test method measures the bulk volume by controlling the consolidation force and measuring the displacement of a plunger used to compact the bed of coke.

## 5. Significance and Use

5.1 The bulk density is an indicator of calcined petroleum coke porosity and packing capability which is an important coke property for anode production in aluminum industry. This procedure will allow an automated measurement of specific sized fractions ranging from 8 mm to 0.25 mm coke particles.

5.2 Results from this test method are used in determining coke specifications, classification purposes, and for quality control.

## 6. Apparatus

6.1 *Balance,* capable of measuring 100 g ± 0.0001 g.

6.2 *Table Top Riffler:*

6.2.1 *Pycnometer,* equipped with a force transducer and suitable 50.8 mm (2 in.) or larger glass chamber and plunger assembly.

[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.

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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.

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

NOTE 1—GeoPyc 1360[3] has been successfully used for this analysis.

6.3 *Wire Mesh Sieves,* 8 in. diameter, round, 8.00 mm (5⁄16 in.), 4.00 mm (No. 5), 2.00 mm (No. 10), 1.00 mm (No. 18), 0.50 mm (No. 35), 0.25 mm (No. 60), pan, and cover.

6.4 *Ro-tap Sieve Shaker,* for 8 in. diameter sieves.

## 7. Sampling, Test Specimens, and Test Units

7.1 Grab a representative sample according to Practice D6970 and reduce the sample to 900 g ± 100 g following Practice D6969.

7.2 Sieve the representative sample using the following screens: 8.00 mm (5⁄16 in.), 4.00 mm (No. 5), 2.00 mm (No. 10), 1.00 mm (No. 18), 0.50 mm (No. 35), and 0.25 mm (No. 60).

7.3 Using a sieve shaker, shake the sieves for 10 min.

7.4 Discard the top sieve material (8.00 mm (5⁄16 in.)) and transfer each sized fraction of calcined petroleum coke into appropriate containers.

## 8. Preparation of Apparatus

8.1 Follow manufacturer's instructions for initial assembly, conditioning, and preparation of pycnometer.

## 9. Calibration and Standardization

9.1 Follow the instructions for the equipment used.

9.2 A blank data set can be performed by following the procedure based upon the equipment used. If the GeoPyc (see Note 1) is used, follow detail in Annex A1 and Table 1.

**TABLE 1 Recommended Equipment Setting**

| | |
|---|---|
| Number of Cycles: | 10 |
| Consolidation Force: | 30 N |
| Preparation Cycles: | 5 |
| Agitation: | High |
| Speed: | 150 steps/s |

9.3 A blank data set shall be performed each time a new cylinder is used or when the apparatus is moved.

## 10. Procedure

10.1 Split the sieved sample fraction to a test specimen of 52 g ± 2 g.

10.2 Weigh the prepared test specimen to 0.0001 g and record the weight.

10.3 Pour the entire test specimen into the glass chamber and insert PTFE plunger.

10.4 Fit the chamber and plunger assembly onto the pycnometer.

10.5 Start the analysis following the procedure in the instruction manual.

10.6 Enter the necessary or required information such as sample identification, sample mass, or other user-selected information.

10.7 Choose the stored blank data set for the analysis.

10.8 Press "Enter." The analysis will begin and will be performed automatically.

10.9 Record the final volume or density when pycnometer has finished analyzing the test specimen. You may also print a copy of the report if a printer is attached.

## 11. Calculation or Interpretation of Results

11.1 The pycnometer measures the volume of each consolidation cycle and then averages the results of all the runs. The average volume and specimen weight are used to calculate the specimen bulk density. The final results are reported in $cm^3$ and $g/cm^3$, with three decimals.

## 12. Report

12.1 The specific size fraction must be reported as part of the test results.

## 13. Precision and Bias

13.1 The precision of this test method is based on an interlaboratory study of D8097, conducted in 2016. Five laboratories tested five unique petroleum coke specimen types/sizes. Every test result represents an individual determination, and all participants reported duplicate test results for each material tested. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. RR:D02-1848.[4]

13.1.1 *Repeatability (r)*—The difference between two independent results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would exceed the following value with an approximate probability of 5 % (one case in 20 in the long run) in the normal and correct operation of the test method.

13.1.1.1 Repeatability limits are listed in Tables 2-6.

$$\text{Repeatability} = 0.016\ g/cm^3 \quad (1)$$

13.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would exceed the following value with an approximate probability of 5 % (one case in 20 in the long run) in the normal and correct operation of the test method.

13.1.2.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility

[3] The sole source of supply of the apparatus known to the committee at this time is Micromeritics Instrument Corporation, 4356 Communications Dr., Norcross, GA 30093, USA. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1848. Contact ASTM Customer Service at service@astm.org.