Designation: D8097 – 17

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

3.1.2 *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.

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.

[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.

[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.

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 of the top sieve material (5⁄16 in. (8.00 mm)) 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 repetitive 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 in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

13.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability

[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.

TABLE 2 Bulk Density (g/cm^3), 0.50 mm by 0.25 mm

Material	Average[A] $\bar{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
Coke A	0.8849	0.0037	0.0065	0.0102	0.0181
Coke B	0.8178	0.0026	0.0103	0.0073	0.0288
Coke C	0.9773	0.0032	0.0136	0.0090	0.0381
Coke D	0.9010	0.0019	0.0064	0.0052	0.0180
Coke E	0.8358	0.0091	0.0107	0.0254	0.0299

[A] The average of the laboratories' calculated averages.

conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

13.1.1.2 Repeatability limits are listed in Tables 2-6.

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

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, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

13.1.2.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

13.1.2.2 Reproducibility limits are listed in Tables 2-6.

$$\text{Reproducibility} = 0.031 \quad \text{g/cm}^3 \quad (2)$$

13.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

13.1.4 Any judgment in accordance with statements 13.1.1 and 13.1.2 would have an approximate 95 % probability of being correct.

13.2 *Bias*—At the time of the study, no accepted reference material suitable for determining the bias for this test method was incorporated, therefore no statement on bias is being made.

13.3 The precision statement was determined through statistical examination of 250 test results, from five laboratories, on five materials. The materials tested were described as:

13.3.1 *Coke A*—Calcined petroleum coke—rotary kiln, anode.

13.3.2 *Coke B*—Calcined petroleum coke—rotary kiln, anode.

13.3.3 *Coke C*—Calcined petroleum coke—vertical shaft, anode.

13.3.4 *Coke D*—Calcined petroleum coke—rotary kiln, anode.

13.3.5 *Coke E*—Calcined petroleum coke—rotary kiln, anode.

13.4 To judge the equivalency of two test results, it is recommended to choose the average for the specified size fraction being analyzed as listed in Table 7.

14. Keywords

14.1 bulk density; calcined petroleum coke; natural fraction; pycnometers; tapped bulk density (TBD); transaxial pressure; vibrated bulk density (VBD)

TABLE 3 Bulk Density (g/cm³), 1 mm by 0.50 mm

Material	Average[A] $\bar{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
Coke A	0.8244	0.0058	0.0096	0.0162	0.0268
Coke B	0.7631	0.0019	0.0094	0.0052	0.0263
Coke C	0.9205	0.0054	0.0118	0.0152	0.0331
Coke D	0.8796	0.0084	0.0090	0.0234	0.0251
Coke E	0.7764	0.0031	0.0087	0.0086	0.0243

[A] The average of the laboratories' calculated averages.

TABLE 4 Bulk Density (g/cm³), 2 mm by 1 mm

Material	Average[A] $\bar{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
Coke A	0.7548	0.0034	0.0075	0.0094	0.0209
Coke B	0.7213	0.0120	0.0192	0.0336	0.0538
Coke C	0.8453	0.0052	0.0148	0.0145	0.0414
Coke D	0.8449	0.0056	0.0094	0.0158	0.0263
Coke E	0.7294	0.0048	0.0099	0.0134	0.0278

[A] The average of the laboratories' calculated averages.

TABLE 5 Bulk Density (g/cm³), 4 mm by 2 mm

Material	Average[A] $\bar{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
Coke A	0.6864	0.0079	0.0114	0.0222	0.0319
Coke B	0.7104	0.0109	0.0143	0.0305	0.0400
Coke C	0.7635	0.0016	0.0114	0.0045	0.0320
Coke D	0.7816	0.0106	0.0108	0.0296	0.0304
Coke E	0.6695	0.0103	0.0154	0.0290	0.0431

[A] The average of the laboratories' calculated averages.

TABLE 6 Bulk Density (g/cm³), 8 mm by 4 mm

Material	Average[A] $\bar{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
Coke A	0.6559	0.0063	0.0080	0.0177	0.0223
Coke B	0.6929	0.0057	0.0149	0.0159	0.0418
Coke C	0.7142	0.0039	0.0102	0.0110	0.0286
Coke D	0.7202	0.0036	0.0117	0.0102	0.0328
Coke E	0.6513	0.0090	0.0124	0.0252	0.0347

[A] The average of the laboratories' calculated averages.

TABLE 7 Average Repeatability and Reproducibility Limit for Each Specified Size Fraction

Specific Size	Average[A] $\bar{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
0.50 mm by 0.25 mm	0.8834	0.0041	0.0095	0.0114	0.0266
1.00 mm by 0.50 mm	0.8328	0.0049	0.0097	0.0137	0.0271
2.00 mm by 1.00 mm	0.7791	0.0062	0.0122	0.0173	0.0340
4.00 mm by 2.00 mm	0.7223	0.0083	0.0127	0.0232	0.0355
8.00 mm by 4.00 mm	0.6869	0.0057	0.0114	0.0160	0.0320

[A] The average of the laboratories' calculated averages.

ANNEX

(Mandatory Information)

A1. BLANK DATA SET

A1.1 Mount the empty glass chamber/plunger assembly into instrument securely.

A1.2 Select the "Store Blank Data" setup.

A1.3 Name the blank data set (1 to 10) by pressing the second key, then "Analyze." Use the arrow keys to choose "Store Blank Data," and then choose a blank data set number (1 to 10).

A1.4 Select (10) for the number of cycles.

A1.5 Enter the diameter of the chamber being used. This diameter will be used for all subsequent runs in this stored blank data set.

NOTE A1.1—If you wish to store data for a different size chamber, you must start another blank data set.

A1.6 Enter the consolidation force being used.

NOTE A1.2—The software prevents you from storing duplicate forces/pressures within a single blank data set.

A1.7 Start analysis by selecting "Enter." The GeoPyc 1360 automatically collects blank data for the selected chamber using the designated force. The following message is displayed: "Press Enter for another blank run, or Esc to end."

A1.8 To continue storing the blank data in this set, repeat this process beginning with the force. To end, press "Escape."