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An American National Standard

Standard Test Method for Determination of Vibrated Bulk Density of Calcined Petroleum Coke[1]

This standard is issued under the fixed designation D 4292; 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 editorially moved into the standard text in March 2003.

1. Scope

1.1 This test method covers the determination of bulk density of a representative 2-kg sample of calcined petroleum coke, after vibration to increase compaction.

1.2 The procedure is limited to particles passing through a 6.68-mm opening sieve (equivalent to a 3-mesh Tyler Standard Series) and retained on a 0.21-mm opening sieve (equivalent to a 65-mesh Tyler Standard Series). Further, the procedure is limited to a specific test sample having particles retained between screens having openings that differ by a factor of less than 2 $\sqrt{2}$ and preferably less than 2.

1.3 The values stated in acceptable SI units are to be regarded as the standard.

1.4 *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:*

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 2013 Practice of Preparing Coal Samples for Analysis[2]

D 2234 Practice for Collection of a Gross Sample of Coal[2]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

E 11 Specification for Wire Cloth and Sieves for Testing Purposes[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *as-calcined particles*, *n—of coke,* those that have not been subject to laboratory crushing.

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

3.1.3 *laboratory crushed particles*, *n—of coke*, those that have been crushed in the laboratory.

4. Summary of Test Method

4.1 After appropriate crushing of the calcined coke, using both the jaw crusher and roll crusher, the test volume of 100 g is measured after vibration and the bulk density is calculated.

5. Significance and Use

5.1 Vibrated bulk density, VBD, is an indicator of calcined petroleum coke porosity, which affects its suitability for use in pitch-bonded carbon applications. (**Warning—**Vibrated bulk density for a sample of calcined petroleum coke is strongly dependent upon average particle size and particle size range. Bulk density tends to increase with decreasing coke size. A narrow particle size range for this test minimizes the possibility for variation due to skewing of the test sample toward either screen defining the sample. Particle size range tested should be agreed upon by the purchaser and supplier.)

NOTE 1—An example of the use of VBD to characterize coke for prebaked anodes for aluminum smelting is reported by Belitskus[5] who found particles passing through a 0.59-mm opening, No. 30, sieve and retained on a 0.30-mm opening, No. 50, sieve to be preferred. Other popular ranges are particles passing through a 2.36-mm opening, No. 8, sieve and retained on a 1.17-mm opening, No. 16, sieve for the continuous Soderberg anode process and particles passing through a 6.68-mm opening sieve (equivalent to a 3-mesh Tyler Standard Series) and retained on a 3.33-mm opening, No. 6, sieve for graphite electrode manufacture.

6. Apparatus

6.1 *Jaw Crusher*, laboratory type; jaw opening, approximately 50 by 200 mm; jaws can be set to gaps of approximately 3.2 to 12.7 mm; manganese steel jaw plates.

[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] *Annual Book of ASTM Standards*, Vol 05.06.

[3] *Annual Book of ASTM Standards*, Vol 05.02.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Belitskus, D. L., "Evaluating Calcined Coke for Aluminum Smelting by Bulk Density," *Aluminium*, Vol 51, No. 2, 1975.

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6.2 *Roll Crusher*, laboratory type; glass hardened rolls; roll diameter, approximately 200 mm; roll width, approximately 150 mm; gap range from 0 to 12.7 mm.

6.3 *Sieve Shaker*, electrical drive with an automatic timer; should have a rotating and tapping action.

6.4 *Sieves*—meeting Specification E 11.

6.5 *Pan Balance*, accurate to 0.1 g, capacity 2.0 kg.

6.6 *Vibrator*[6], with approximately 175- by 250-mm deck, must be capable of vibrating at a frequency of 60 Hz and an amplitude of 0.20 to 0.22 mm (peak) when loaded with a 50-g cork ring, 215-g graduated cylinder, and a 100-g coke sample.

6.7 *Ohmmeter*, adequate to test continuity of an electrical circuit.

6.8 *Cork Ring*, approximately 100-mm inside diameter by 25 mm high by 12 mm thick, weight approximately 50 g (round-bottom flask support).

6.9 *Graduated Cylinder*, glass, 250 mL, inside diameter approximately 37 mm, base diameter approximately 95 mm.

6.10 *Plastic Funnel*, must have a stem with straight sides and an outside diameter of 25 to 30 mm (powder funnel).

6.11 *Automatic Timer, Clock, or Watch*, with a second indicator.

6.12 *Riffle Sampler*, enclosed drawer, approximately 380 by 290 by 360 mm, 24-slot.

7. Precautions

7.1 Exercise care in the operation of the jaw crusher and roll crusher. Turn power off at the source when setting the gap. Wear safety glasses and keep hands clear when feeding material. Turn power off at the source when equipment is opened for cleaning after the grinding operation.

8. Sample Preparation

8.1 Use the crushing procedure in 8.2 and subsequent paragraphs so that contributions to VBD from both *as-calcined* and *laboratory-crushed* particles (which differ significantly in density) are included.

NOTE 2—Because the vibrated bulk density method is based on the packing of sized particles, the method of sample preparation can affect results due to differences in particle shapes affecting packing characteristics.

8.1.1 Air-dry the laboratory sample, if it appears to be wet, prior to crushing to avoid caking.

NOTE 3—On agreement by purchaser and supplier, density of only *as-calcined* particles in the selected size range are determined. If so, proceed to Section 11 and report as part of the result that only *as-calcined* particles were used.

NOTE 4—Recommended practice for collecting samples and the equipment and procedures for dividing are described in Test Methods D 346, D 2013, D 2234, and D 4057.

8.2 *Jaw Crusher Operation*—Use the procedure appropriate to the crusher being used, adjust the jaws so that the gap between them (at their closest position to each other in the crushing cycle) is approximately 5 mm. Turn on the jaw crusher motor, slowly feed the sample through the crusher, and collect the product for further reduction through a roll crusher.

8.3 *Roll Crusher Operation*—(**Warning—**To avoid damage to the rolls, size reduction with the roll crusher must be limited to a maximum ratio of 4 to 1. Depending on the fraction desired, a one-step reduction is often not possible from the maximum particle size in the jaw crusher product and intermediate roll settings are used. The sample is reduced to the desired mesh size using as few intermediate settings as possible (but not exceeding the 4 to 1 reduction ratio).

8.3.1 With the motor deactivated, and using a method appropriate to the roll crusher being used, adjust the roll gap according to the following procedure. If the rolls are readily accessible, adjustment with a leaf-type feeler gage inserted between the rolls with the motor deactivated is useful.

8.3.2 Calculate the ratio of the maximum particle size of the roll crusher feed (expressed as the opening, in millimetres, of the finest screen through which the largest particles will pass) to the maximum particle size of the bulk density fraction required (expressed as the opening, in millimetres, of the coarser of the two screens used to define the bulk density fraction).

8.3.3 Select the number of crushing steps required from the following table:

Ratio	Number of Crushing Steps Required
1.1 to 4.0	1
4.1 to 16.0	2
16.1 to 64.0	3

8.3.4 For each crushing step required, the roll gap is decreased (from a value equivalent to the maximum particle size of the feed) by a factor of:

$$\sqrt[n]{\text{Ratio}} \text{ (as defined in 8.3.2)} \quad (1)$$

where:

n = number of crushing steps required (8.3.3)

8.3.5 For example, it is desired to reduce a coke having a maximum particle size of 6.68 mm to one having a maximum particle size of 0.208 mm. The calculation is as follows:

Ratio = 32.115 (see 8.3.2)

Crushing steps required = 3 (see 8.3.3)

Factor = $\sqrt[3]{32.115}$ = 3.179 (see 8.3.4)

1st setting: 6.68 mm ÷ 3.179 = 2.101 mm

2nd setting: 2.101 mm ÷ 3.179 = 0.661 mm

3rd setting: 0.661 mm ÷ 3.179 = 0.208 mm

8.3.6 After the roll gap is adjusted, remove the feeler gage (if used), turn on the roll crusher motor, slowly feed 0.3 kg of the jaw crusher product through the roll crusher, and collect the sample. When more than one roll crushing step is required, regrind through smaller openings and collect the sample. Then, using the appropriate screens (those defining the bulk density fraction), sample receiver, and cover, sieve the roll-crushed sample in the sieve shaker. With this final roll crusher setting, at least 30 % of the coke generally will be in the desired particle size range.

8.3.7 This setting will produce roughly equal weights of coke coarser and finer than the desired fraction, provided that the starting material is sufficiently coarse. If yield is at least 30 % and the ratio of coarser to finer product is between 0.8

[6] The calibration procedure described later is specific to a Syntron Model J-1A or J-1B Jogger (from FMC Corp., Material Handling Equipment Div., Homer City, PA). Statistical data were obtained using Model J-1A Joggers.

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