Designation: D4292 – 92 (Reapproved 2007)

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

This standard is issued under the fixed designation D4292; 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 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:*[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

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

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[3] 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.

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.

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

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

6.4 *Sieves*—meeting Specification E11.

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

6.6 *Vibrator*[4], 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 D346, D2013, D2234/D2234M, and D4057.

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 and 2.0, crushing is satisfactory and the remainder of the material is fed through the roll crusher, using as many

[4] 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.

intermediate settings as required. The entire roll crusher product is consolidated and the desired fraction separated.

8.3.8 At the roll gap setting intended to maximize the final product, proceed as follows if the criteria in 8.3.7 are not met. If after one pass the ratio of coarser to finer-than-desired coke is greater than 2.0, decrease the roll gap to 80 % of its original value and test another 0.3 kg sample of jaw crusher product or intermediate roll crusher product, if required. (If the product is just slightly too coarse, an alternative procedure is to make multiple passes through the roll crusher with the original gap setting.)

8.3.9 If the ratio is less than 0.8 and the starting material is coarse enough to permit this ratio to be attained, increase the roll gap by 20 % of its original value and retest with 0.3 kg of jaw crusher product or intermediate roll crusher product, if required. If the starting material is not coarse enough to obtain this ratio for the particle size range selected for the test, disregard this restriction.

8.3.10 These procedures will result in a satisfactory sample as defined in 8.3.7 in the majority of cases. If not, adjust roll settings or make multiple passes, or both, with trial-and-error adjustments until a satisfactory composite sample is obtained from the 2-kg starting sample. Observe that 0.3-kg fractions of the jaw crusher product can be discarded in their entirety if too fine after roll crushing. Partial consolidation of roll crusher products is not acceptable; that is, once a 0.3-kg sample of jaw crusher product has been passed through the roll crusher, it must either be consolidated in its entirety with other roll crusher products or discarded. A minimum of 210 g of properly sized vibrated bulk density sample is required.

9. Preparation of Apparatus

9.1 *Graduated Cylinder*—Since vibrator amplitude is affected by weight on the table, cut off the graduate below the pouring lip so that the weight is 215 ± 10 g. Sand sharp edges. With one common brand of graduated cylinder, this corresponds to an overall length of about 305 mm. Fit the graduate with a No. 8 stopper (tight fit).

9.2 *Vibrator*—Fasten the cork ring securely with screws to the table top of the vibrator as a retainer ring for the graduated cylinder during the test. (Drill and tap holes in the vibrator table as required.) The inner diameter of the cork ring is intentionally larger than the base width of the graduated cylinder. It is designed only to keep the graduated cylinder from vibrating off the vibrator table.

10. Calibration of Apparatus

10.1 *Graduated Cylinder Calibration Factor*—Measure the distance, in millimetres, between the 90-mL and 170-mL marks on the graduated cylinder.

$$B = 170 - 90/\text{distance in mm} \quad (2)$$

where:

B = calibration factor, mL/mm.

Wrap a piece of masking tape around the graduate with the 190-mL mark at the bottom of the tape. Make eight marks on the bottom of the tape equidistantly around the cylinder. These are used only for equally spacing height measurements (not as a volume measurement base line).

10.2 *Determination of Accuracy of Graduated Cylinder*—Using distilled water at 25 ± 5°C, fill the graduate to the 90-mL mark and determine the weight of water at that volume. Using a density of 0.997 g/cm^3 for water, determine the true volume. Do the same at 20-mL increments up to the 170-mL mark. If the deviation at any indicated volume is greater than ±0.5 mL, a table of indicated volumes versus true volumes should be made for use in computing bulk density.

10.3 *Vibrator*[5]—Using a hex wrench, remove the four screws that hold the vibrator table and remove the table. Loosen the core locking screw. If the aluminum table is grounded, remove the ground wire. Turn the vibrator on its side and attach the ohmmeter to the table support and ground. Turn the core into the case (clockwise) until the ohmmeter registers zero (core is touching armature). Back off the core (counter-clockwise) until the ohmmeter reads infinite resistance. Mark the zero position with arrows on each side of the screw slot and turn the core out exactly one and three-eighths turns. Replace the table and tighten the four screws.

11. Procedure

11.1 Weigh 100.0 ± 0.1 g of the coke fraction to be tested into a container. Pour the coke *slowly* through a funnel into the graduate. Transfer time must be 70 to 100 s. The importance of this step on the density value cannot be overemphasized. A rapid pour results in a higher volume than a slow pour and a part of the difference in volume is retained through the vibrating step.

11.2 Fit the rubber stopper tightly to the top of the graduate.

11.3 Place the graduate inside the retaining ring on the vibrator.

11.4 Vibrate for 5 min at a dial setting giving an amplitude of 0.20 to 0.22 mm (peak) at 60 Hz.

NOTE 5—Although the statistical data in Section 14 were obtained using a 5-min vibration time, a subsequent test at one laboratory on 8 coke samples resulted in an average vibrated bulk density only 0.022 g/cm^3 lower after 1 min of vibration than after 5 min of vibration. For routine use of this test method within a laboratory, the shorter time may be preferred.

11.5 *Measurement of Compacted Volume*:

11.5.1 Choose and record a line on the graduate below the top of the compacted sample column from which to measure the height of the sample. This will be known as the base line. For example, if the top of the sample column is near the 100-mL mark, the 90-mL mark may be chosen for the base line.

11.5.2 If deemed necessary (see 10.2), consult the table of indicated volume versus true volume and use the true volume at the base line.

11.5.3 Using a small metric rule, measure the distance from the base line to the top of the compacted column at eight points around the graduate. These distances are measured to the nearest 0.5 mm. Record and average the measurements.

[5] This calibration procedure is for a Syntron Model J-1A or J-1B Jogger only. With a weight equivalent to the total weight of the cork ring, graduate, and test sample attached to the vibrator table, the procedure results in a vibration amplitude of 0.20–0.22 mm (peak) at a fixed 60 Hz at a dial setting of *5* for Model J-1A or *35* for Model J-1B. To be satisfactory, other vibrator models must be able to be calibrated to produce these vibration parameters.

11.6 Repeat the procedure, beginning at 11.1, with a second 100.0-g sample.

12. Calculations

12.1 Calculate the volume as follows:

$$\text{Volume, mL} = (A \times B) + C \quad (3)$$

where:

A = average sample height from base line, mm,
B = calibration factor, mL/mm, and
C = base line, mL, corrected for graduated cylinder error, if necessary (see 10.2).

$$\text{VBD, g/cm}^3 = \frac{\text{weight in grams}}{\text{volume in millilitres}} \quad (4)$$

Average the values for the two determinations.

13. Report

13.1 Report the average of the two determinations to the nearest 0.001 g/cm^3. The particle size range must be reported as part of the test results. For example, VBD (−30 + 50 sieve size) = 0.890 g/cm^3.

14. Precision and Bias [6]

14.1 Precision was determined by interlaboratory testing of calcined petroleum coke samples crushed and sized by the participants to pass through a 0.83-mm opening, (No. 20 sieve) and be retained on a 0.30-mm opening, (No. 50 sieve). Statistical information was calculated in accordance with methods outlined in D02-1007.

NOTE 6—Precision for vibrated bulk density on other ranges has not been determined.

14.2 *Precision*—The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows.

14.2.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions 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 twenty.

$$\text{Repeatability} = 0.014 \text{ g/cm}^3 \quad (5)$$

14.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in one case in twenty.

$$\text{Reproducibility} = 0.046 \text{g/cm}^3 \quad (6)$$

14.3 *Bias*—This test method is empirical and no statement as to bias is made.

15. Keywords

15.1 calcined petroleum coke; porosity; vibrated bulk density

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1166.