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Standard Test Method for Determination of Vibrated Bulk Density of Calcined Petroleum Coke using a Semi-Automated Apparatus[1]

This standard is issued under the fixed designation D7454; 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, using a semi-automatic apparatus.

1.2 The procedure is applied, but not limited, to particles passing through a 4.75-mm opening sieve and retained on a 1.18-mm opening sieve. Further, the procedure is applied, but not limited, to a specific test sample having particles passing through a 0.85-mm opening sieve and retained on a 0.425-mm opening sieve. This procedure could also be applied to other sieve fractions being agreed on in the aluminum industry as specified in Annex A1.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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]

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 *Definitions:*

3.1.1 *as-calcined particles*, *n—of coke*, those particles 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 particle size range to the volume occupied.

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

4. Summary of Test Method

4.1 The natural 4.75 by 1.18-mm fraction of the original coke is separated from the sample by manual screening, ground to 0.85 by 0.425 mm, and fed at a controlled rate into a graduated cylinder on a vibrating table until the coke reaches the 50-mL mark. The collected coke is weighed and the bulk density is calculated and reported in g/mL.

4.2 The procedure is empirical; close adherence to the technique and apparatus is necessary to ensure reproducible results. To provide comparable results in different locations, exact adjustments of operating parameters are required using reference samples.

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.)

6. Apparatus

6.1 *Pan Balance*—Accurate to 0.1 g, with a capacity of 2.0 kg.

6.2 *Riffle Sampler*—Enclosed drawer, approximately 380 by 290 by 360 mm, 24-slot.

6.3 *Sieves*—Meeting Specification E11.

6.4 *Sieve Shaker*—Electrical drive with an automatic timer; should have a rotating and tapping action.

6.5 *Roller Crusher*—Laboratory type; glass hardened rolls; roll diameter of approximately 150 mm; roll width of approximately 150 mm; gap range from 0 to 12.7 mm.

6.6 *Thickness Gauges (leaf-type)*—0.4, 1.0, 1.5, and 4.0 mm.

6.7 *Semi-Automated VBD Apparatus*, As shown in Fig. 1. See also comments about installation in Annex A1.

[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.

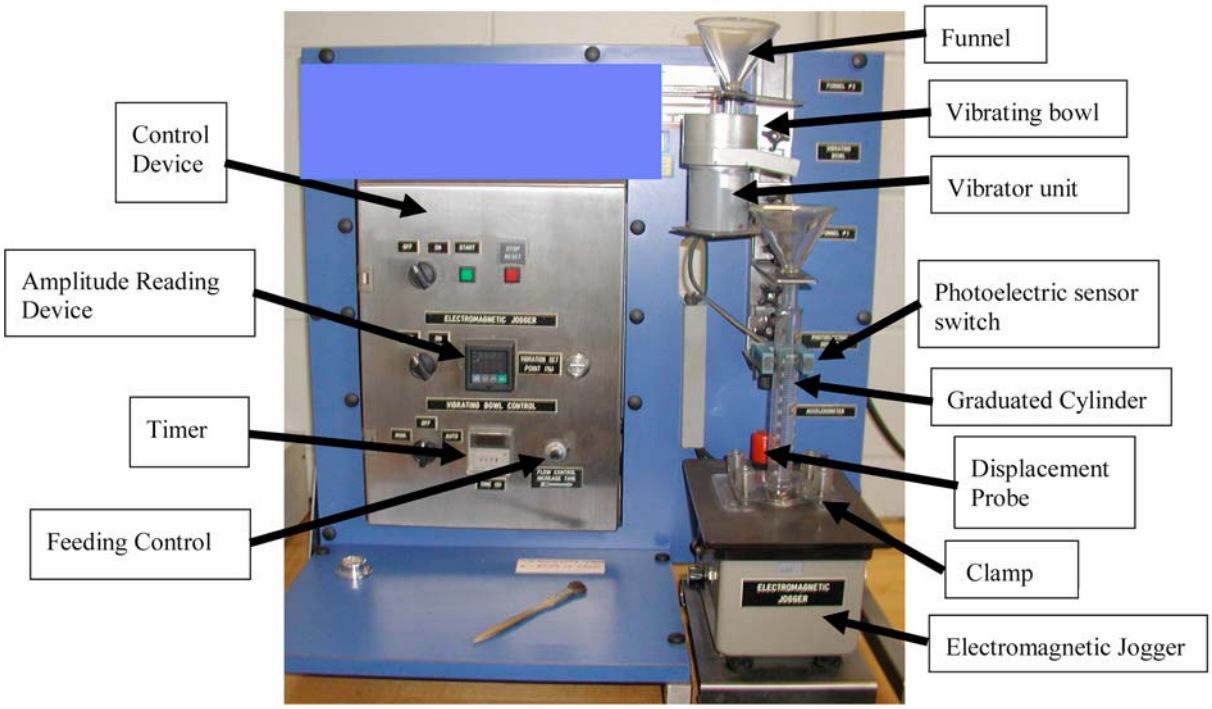

FIG. 1 Example of Semi-Automated Apparatus Set-Up

6.7.1 *Borosilicate Glass Powder Funnels*—8-cm diameter funnels with 1-cm internal diameter and stems about 3.5 cm long. Tips of funnels should be cut at a right (not oblique) angles (see Fig. 1). The distance between the tip of the upper funnel and the bottom of the vibrating bowl should be around 6 mm.

6.7.2 *Electromagnetic Jogger*—With approximately 175- by 250-mm deck, and shall be capable of vibrating at a frequency of 60 Hz.

6.7.3 *Acrylic Clamp*—To hold cylinder.

6.7.4 *Vibrating Bowl*—Having a diameter of approximately 7.5 cm and a height of 4.0 mm, such as that being used with rotary micro riffler.

6.7.5 *Displacement Probe and Reading Device*—Permitting continuous monitoring of amplitude vibration.

6.7.6 *Graduated Cylinder*—50 mL, with inside diameter approximately 23 mm and height approximately 19 cm.

6.7.7 *Photoelectric Sensor Switch*.

6.7.8 *Control Device*—Permitting real-time adjustment of the vibration amplitude and automatic stopping of the feeding device when the coke level reaches the 50-mL mark.

6.7.9 *Automatic Timer, Clock, or Watch*—With a second indicator.

6.7.10 *Line Stabilizer (Optional)*—Use if the noise on the power line is significant and affects the apparatus performance.

6.7.11 *Round Level*.

6.7.12 *Balance*—0 to 300 g and sensitive to 0.01 g.

7. Hazards

7.1 Exercise care in the operation of the roll crusher.

7.1.1 Wear safety glasses and keep hands clear when feeding material.

7.1.2 Turn power off at the source when equipment is opened for cleaning after the grinding operation.

8. Sample Preparation

8.1 Reduce the original sample volume to about 1 kg.

8.2 Manually screen out the natural to 4.75 by 1.18 mm and < 1.18 mm.

8.3 Transfer the 4.75 by 1.18-mm fraction into a suitable plastic bag and homogenize manually.

8.4 Weigh 180 to 200 g of 4.75 by 1.18 mm material.

8.5 Using the Starrett thickness gauges, adjust roller spacing to 4.0 mm. Slowly feed the roller crusher with the 4.75 by 1.18-mm fraction by spreading the material all over the rollers.

8.6 Adjust the spacing between rollers to 1.5 mm to regrind the material. Set the spacing between the rollers to 1.0 mm and regrind the material a second time.

8.7 Manually screen out the 0.85 by 0.425-mm fraction and transfer it into a plastic bag. Discard the < 0.425-mm fraction and keep the > 0.85-mm fraction.

8.8 Adjust the roller spacing to 0.5 mm and grind the > 0.85-mm fraction. Manually screen out the 0.85 by 0.425-mm fraction and add this fraction into the same plastic bag referred to in 8.7. Discard the < 0.425 mm material and recuperate the > 0.85-mm fraction, if present.

8.9 Repeat, if necessary, the grinding procedure in 8.8 of the > 0.85-mm fraction, until all particles pass through the 0.85-mm sieve. It is possible that at this step, about 1 to 3 g of particles larger than 0.85 mm cannot be ground to finer particles. Do not attempt to grind them using roller spacing smaller than 0.5 mm. Simply discard them (these particles are in general, plate-like shape particles and should not be used for bulk density measurement).

8.10 Manually mix the contents of the plastic bag.

8.11 Divide the 0.85 by 0.425-mm material between two sets of sieves with openings of 0.85 mm and 0.425 mm and their pan. Using a sieving shaker screen out the 0.85 by 0.425-mm fraction for 7 min. Discard the < 0.425 mm material.

8.12 Transfer the 0.85 by 0.425-mm material into an appropriate plastic container and manually mix the contents (about 100 mL of material is needed for analysis).

9. Preparation of Apparatus

9.1 Install the apparatus as shown Fig. 1.

10. Calibration and Standardization

10.1 *Calibration of Graduated Cylinder*—Adjust the height of the photodetector, and determine the true volume at the 50-mL mark of the graduated cylinder, following the detailed procedure given in Annex A2. Calibration shall be made each time a new cylinder is used or when the apparatus is moved.

10.2 *Determination of the Displacement Speed Target of the Jogger*—Determine the displacement target, in accordance with Annex A3, using standard reference materials. Once established, this target shall be kept indefinitely unless the probe, the controller or the jogger have to be changed.

10.3 *Feeding Rate*—Check/adjust the feeding rate for each sample.

11. Procedure

11.1 Make sure that the vibrating table is levelled.

11.2 Turn on the apparatus at least 10 min before initiating measurements. The power should not be turned off between readings.

11.3 Weigh the graduated cylinder to the nearest 0.01 g, insert it into the clamping device on the vibrating table while ensuring it does not touch the photoelectric sensor, and let the table vibration stabilize to the set points (it takes a few seconds).

11.4 Fill the upper funnel with the coke sample.

11.5 Fill the cylinder to about half, using maximum feeding rate, to make a constant bed in the vibrator bowl. Stop the feeding, empty the cylinder in the upper funnel, and reinsert it in the clamping device.

11.6 Using the automatic mode, reset the chronometer, initiate the feeding and adjust if necessary, the bowl vibration intensity to obtain the feeding time of 30 ± 3 s/10 mL. The feeding will automatically stop when the 50-mL mark is reached. If no feeding time adjustment was necessary and if the discharge time falls within 135 and 165 s, proceed to 11.7. Otherwise, repeat 11.6 until the time target is met.

11.7 Empty the cylinder in the upper funnel, reinsert it into the clamping device, and let the table vibration stabilize to the set points (it takes few seconds). Then, using the automatic mode, reset the chronometer and initiate feeding. The feeding will stop automatically.

11.8 Check that the discharge time falls within 135 and 165 s (150 ± 15 s). If not, return the coke to the upper funnel and repeat from 11.6 to readjust the feeding time.

11.9 Remove the cylinder containing the coke and weigh to the nearest 0.01 g. Take two additional readings according to 11.7, readjusting, if necessary, the feeding time to meet the target (between 135 and 165 s).

11.10 If the difference between the lowest and highest weight readings exceeds 0.40 g, check if the apparatus is properly functioning, and repeat the test until two consecutive runs agree within the specified 0.40 g. Discard readings only if a malfunction was identified (for example, the apparatus was not on speed displacement target, or the time target was not met).

12. Calculation or Interpretation of Results

12.1 Calculate the average of all acceptable weight readings (at least three).

12.2 Calculate VBD using the following equation:

$$\text{Vibrated Bulk Density (g/mL)} = \frac{\text{Average weight of coke (g)}}{\text{Calibrated volume of cylinder (mL)}} \quad (1)$$

13. Report

13.1 Report the average of the readings to the nearest 0.001 g/mL.

14. Precision and Bias

14.1 *Precision*—The repeatability standard deviation has been determined to be 0.0036 g/mL by making eight independent determinations (0.864, 0.860, 0.854, 0.859, 0.864, 0.859, 0.864, 0.863 g/mL) for a same sample within a very short period of time. The reproducibility of this test method will be made available on or before December 2009.

14.2 *Bias*—This test method is empirical; no statement as to bias is made.

15. Keywords

15.1 calcined petroleum coke; porosity; vibrated bulk density

ANNEXES

(Mandatory Information)

A1. OTHER SIEVE FRACTIONS USED IN THE ALUMINUM INDUSTRY TO DETERMINED VIBRATED BULK DENSITY

A1.1 From a 2 kg as-received sample, split the test sample with a riffle sampler to a subsample of 350 ± 50 g.

A1.2 Sieve the subsample to collect the as-received particle fraction to be measured in accordance with Table A1.1. Discard the undersized particle fraction.

A1.3 Taking care to avoid over–crushing, crush the over-sized fraction of the subsample in a jaw crusher (of the laboratory type, having manganese steel jaws capable of being set to gaps of approximately 3 to 15 mm) or roll crusher, and place the material between the appropriate two sieves with a pan on the bottom and a lid on the top. Gently agitate the sieves by hand but vigorously enough to collect the crushed particle fraction to be measured and discard the undersized material. Repeat until at least 90 % (and preferably more) of the subsample passes through the upper size sieve for the particle fraction to be measured.

TABLE A1.1 Sieve Fractions

Types of Coke	Nominal Width of Smaller Screen Opening (mm)	Nominal Width of Bigger Screen Opening (mm)
Coke for prebaked anodes	0.25	0.5
Coke for Søderberg anodes	1	2
Coke for cathode blocks	2	4

A1.4 Mix the as-received and crushed particle fractions. A minimum of 110 g of sieved, crushed and mixed particles are required for the VBD test. The mixed sample is sieved for 10 min in a sieve shaker. Discard undersized material.

A2. ADJUSTMENT OF THE PHOTOELECTRIC SENSOR HEIGHT AND GRADUATED CYLINDER CALIBRATION

A2.1 Ensure that the table is level, and turn the apparatus on.

A2.2 Insert the cylinder into the clamping device of the VBD apparatus while ensuring it does not touch the photoelectric sensor.

A2.3 Fill the upper funnel with a typical coke sample.

A2.4 Make sure the electromagnetic jogger is turned on, fill the cylinder approximately up to the 45-mL line using maximum feeding rate, and stop the feeding.

A2.5 Using the automatic mode, alternately start and stop the feeding whenever necessary to bring the coke level to the desired position (should be the 50-mL line of the cylinder).

A2.6 Bring the photoelectric sensor very slightly above the desired position by sliding the bracket (the knob should be unscrewed) while holding the sensor (do not forget to screw the knob afterward).

A2.7 Set the vibrating bowl control to OFF to avoid feeding. Reset the chronometer, press START to get the chronometer running, and adjust the photoelectric sensor height very slowly until it stops the chronometer.

A2.8 Remove the cylinder, transfer the coke back into the upper funnel and reinsert it into the clamping device. Using the automatic mode, reset the chronometer and press start the feeding. The feeding will automatically be stopped by the photoelectric sensor. If necessary, make a fine readjustment of the height of the sensor. Repeat A2.8 until the desired position is reached (should be the 50-mL line).

A2.9 Once the photoelectric sensor is at the desired position, empty the vibrating bowl.

A2.10 Set the vibrating bowl control to OFF.

A2.11 Make sure the electromagnetic jogger is turned OFF.

A2.12 Clean the graduated cylinder, and tare it.

A2.13 Fill the cylinder approximately up to the 48-mL line with distilled water equilibrated to room temperature. Make sure no drops stick to the wall of the cylinder.

A2.14 Insert the cylinder into the clamping device.

A2.15 Reset the chronometer to zero.

A2.16 Start the automatic controller and add distillate water drop-wise until the photoelectric sensor stops the chronometer (a “click” is then heard). Make sure no drops stick to the wall of the cylinder and the meniscus is flat. (If the meniscus is not flat, the cylinder may not be clean enough. Reject this test, empty the cylinder, and repeat from A2.12.

A2.17 Remove the cylinder from the clamping device and weigh it.

A2.18 Measure the water temperature in the cylinder.

A2.19 Empty the cylinder.

A2.20 Repeat A2.14 to A2.19 *nine* other times; record the weight of water each time.

A2.21 Using the appropriate water density (see Table A2.1), calculate the volume corresponding to each measured water mass:

$$\text{Volume (mL)} = \frac{\text{Weight of water (g)}}{\text{Density of water at temperature of measurement (g/mL)}} \quad \text{(A2.1)}$$

A2.22 Calculate the average of the obtained volumes (in A2.21). This average becomes the true volume of the graduated cylinder at the 50-mL mark.

A2.23 Empty and dry the cylinder to be ready for measurement.

TABLE A2.1 Density (Corrected for Buoyancy) of Water at Different Temperatures[A]

Temperature, °C	Density, g/mL
20	0.99718
21	0.99697
22	0.99675
23	0.99652
24	0.99628
25	0.99603
26	0.99576
27	0.99549
28	0.99521
29	0.99493
30	0.99463

[A] *Standard Density and Volumetric Tables*, Circular from Bureau of Standards (now called NIST), No. 19, 6th edition, 1924, p. 53.

A3. DETERMINATION OF THE DISPLACEMENT SPEED TARGET

A3.1 Make sure that the apparatus is well installed and that the vibrating table is level. The height of the photo sensor should have been adjusted and the graduated cylinder should have been calibrated.

A3.2 Turn on the apparatus at least 10 min before initiating the measurements; the power should not be turned off between readings.

A3.3 Make sure that the table controller is in automatic mode.

A3.4 Set the vibration amplitude to any starting value.

A3.5 Using a standard reference material, take measurements in accordance with Section 11.

A3.6 If the difference between the measured value and the assigned value is larger than the uncertainty on the assigned value, increase or decrease the displacement target depending if the measured value is respectively larger or smaller than the assigned value.

A3.7 Repeat A3.5 and A3.6 until the reading agrees with the assigned value within the uncertainty.

A3.8 Once the target is established, take *twelve* consecutive readings to confirm that the target is correct and to check for the performance of the apparatus (standard deviation should be ≤ 0.003).

A3.9 The target should stand indefinitely, unless the displacement probe or the controller or the vibrating table is replaced.