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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.

~~ε1 NOTE—Research Report information was added editorially to the Precision and Bias section in September 2014.~~

1. Scope*

1.1 This test method covers the determination of bulk density of a representative ~~2 kg~~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~~4.75 mm opening sieve and retained on a ~~1.18 mm~~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~~0.85 mm opening sieve and retained on a ~~0.425 mm~~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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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]

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

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

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

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

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, 0.1 g, with a capacity of 2.0 kg.

6.2 *Riffle Sampler*—Enclosed drawer, approximately 380 380 mm by 290 by 360 mm, 24-slot. 290 mm 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; 150 mm; roll width of approximately 150 mm; 150 mm; gap range from 00 mm to 12.7 mm. 12.7 mm.

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

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

6.7.1 *Borosilicate Glass Powder Funnels*—8-cm 8 cm diameter funnels with 1-cm 1 cm internal diameter and stems about 3.5 cm 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 mm.

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

6.7.3 *Acrylic Clamp*—To hold cylinder.

6.7.4 *Vibrating Bowl*—Having a diameter of approximately 7.5 cm 7.5 cm and a height of 4.0 mm, 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, 50 mL, with inside diameter approximately 23 mm 23 mm and height approximately 19 cm. 19 cm.
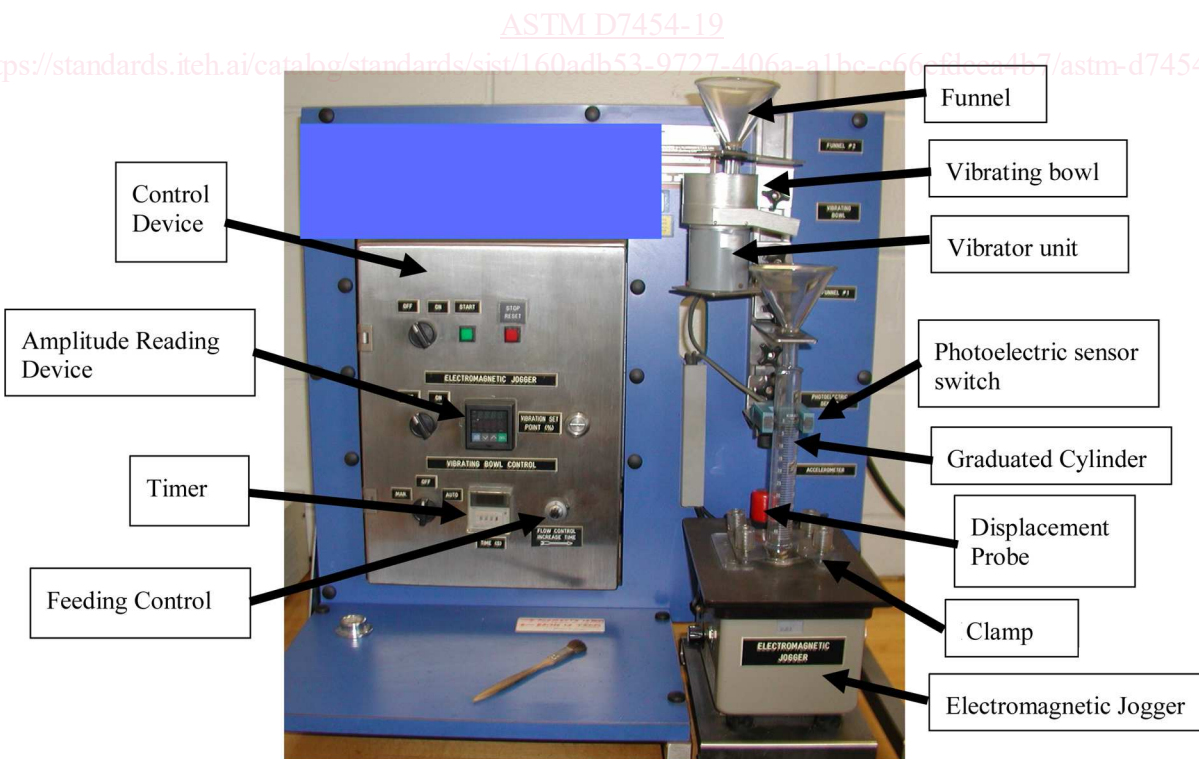
FIG. 1 Example of Semi-Automated Apparatus Set-Up

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~~ 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~~0 g to ~~300 g~~ 300 g and sensitive to ~~0.01 g.~~ 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.~~ 1 kg.

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

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

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

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

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

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

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

8.9 Repeat, if necessary, the grinding procedure in 8.8 of the ~~> 0.85-mm~~> 0.85 mm fraction, until all particles pass through the ~~0.85-mm~~0.85 mm sieve. It is possible that at this step, about ~~1~~1 g to ~~3 g~~3 g of particles larger than ~~0.85 mm~~ 0.85 mm cannot be ground to finer particles. Do not attempt to grind them using roller spacing smaller than ~~0.5 mm.~~ 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~~0.85 mm by ~~0.425-mm~~0.425 mm material between two sets of sieves with openings of ~~0.85 mm and 0.425 mm~~0.85 mm and 0.425 mm and their pan. Using a sieving shaker screen out the ~~0.85~~0.85 mm by ~~0.425-mm~~0.425 mm fraction for ~~7 min.~~ 7 min. Discard the ~~< 0.425 mm~~< 0.425 mm material.

8.12 Transfer the ~~0.85~~0.85 mm by ~~0.425-mm~~0.425 mm material into an appropriate plastic container and manually mix the contents (about ~~100 mL~~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~~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~~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,~~ 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~~30 s ± ~~3~~3 s ~~s/10 mL.~~ /10 mL. The feeding will automatically stop when the ~~50-mL~~50 mL mark is reached. If no feeding time adjustment was necessary and if the discharge time falls within ~~135~~135 s and ~~165 s,~~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~~135 s and 165 s ~~(150~~(150 s ± ~~15 s).~~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.~~0.01 g. Take two additional readings according to 11.7, readjusting, if necessary, the feeding time to meet the target (between ~~135~~135 s and ~~165 s).~~165 s).

11.10 If the difference between the lowest and highest weight readings exceeds ~~0.40 g,~~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.~~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:

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

13. Report

13.1 Report the average of the readings to the nearest ~~0.001 g~~0.01 g/mL.

14. Precision and Bias[3]

14.1 *Precision*—Precicion was determined by interlaboratory testing of calcined petroleum coke samples. Samples covers the density range included between ~~0.78~~0.78 g/mL and ~~0.94~~0.94 g ~~g/mL.~~ /mL. Each sample were prepared by participants and statistical treatment of data was performed in accordance with Practice E691.

14.2 *Repeatability*—With a confidence limit of 95 %, the difference between consecutive results obtained with the same operator, the same machine within the same day under constant conditions do not exceed the following value.

~~Repeatability (r) = 0.02 g/mL~~

$$Repeatability~(r) = 0.02~g/mL$$

14.3 *Reproducibility*—With a confidence limit of 95 %, the difference between two single and independent analysis, performed by different operators, with different machines, in different laboratories on identical test material would not exceed the following value:

~~Reproducibility (R) = 0.02 g/mL~~

$$Reproducibility~(R) = 0.02~g/mL$$

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

15. Keywords

15.1 calcined petroleum coke; porosity; vibrated bulk density