Designation: D7454 – 14ε1

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.

ε[1] 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 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

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 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.

[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.

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

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.

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

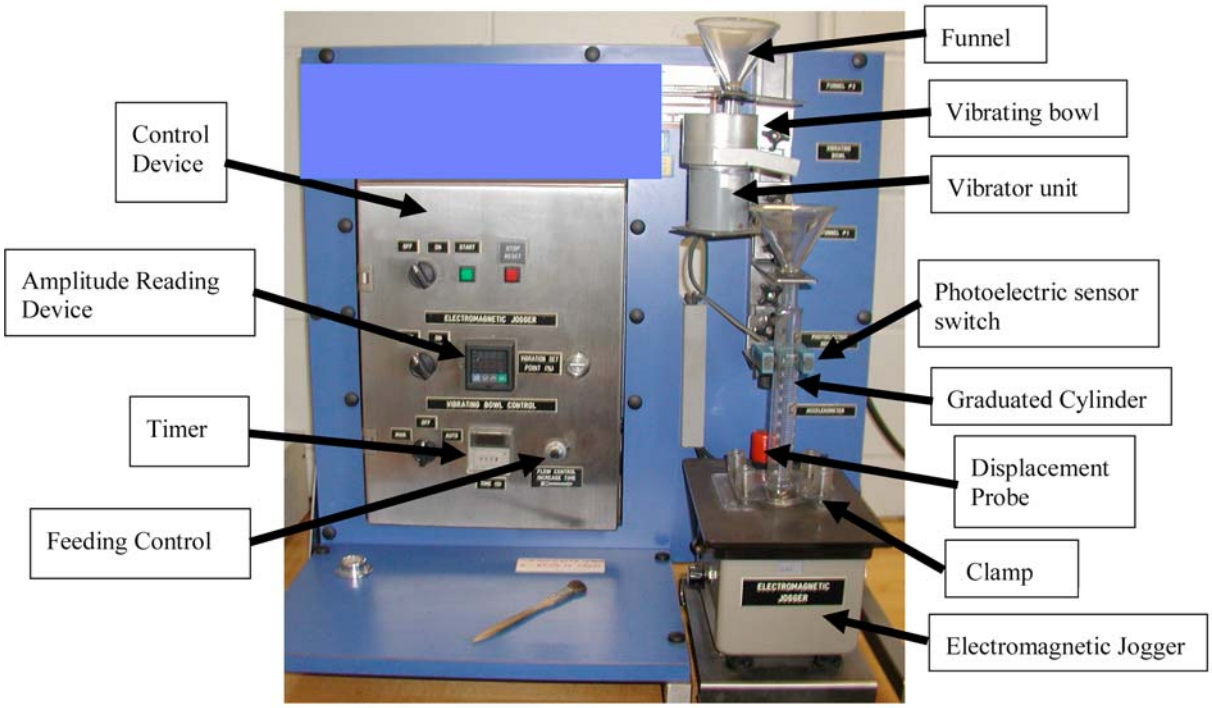

FIG. 1 Example of Semi-Automated Apparatus Set-Up