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Standard Test Method for Ball-Pan Hardness of Activated Carbon¹

This standard is issued under the fixed designation D3802; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers a procedure for determining the ball-pan hardness number of granular activated carbons. For the purpose of this test, granular activated carbons are those having particles 90 % of which are larger than 80 mesh (180 μm) as determined by Test Method D2862.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.3 *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

- B19 Specification for Cartridge Brass Sheet, Strip, Plate, Bar, and Disks
- B150/B150M Specification for Aluminum Bronze Rod, Bar, and Shapes
- D2652 Terminology Relating to Activated Carbon
- D2854 Test Method for Apparent Density of Activated Carbon
- D2862 Test Method for Particle Size Distribution of Granular Activated Carbon
- D2867 Test Methods for Moisture in Activated Carbon
- E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
- E300 Practice for Sampling Industrial Chemicals

3. Terminology

3.1 *General*—Terms applicable to this standard are defined in Terminology D2652.

¹ This test method is under the jurisdiction of ASTM Committee D28 on Activated Carbon and is the direct responsibility of Subcommittee D28.04 on Gas Phase Evaluation Tests.

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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.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *nominal particle size: natural, granular, and irregularly shaped particle carbons*—that particle size range, expressed in terms of Specification E11 sieve sizes, whose small end excludes not more than 5 % of the particle size distribution, and whose large end excludes not more than 5 % of the distribution, on a weight basis.

3.2.2 *nominal particle size: pelleted carbons*—that particle size range, expressed in terms of Specification E11 sieve sizes, whose small end excludes not more than 10 % of the particle size distribution, and whose large end excludes not more than 5 % of the distribution, on a weight basis.

3.2.3 *small end nominal particle size*—that particle size, expressed by its equivalent Specification E11 sieve, which defines the excluded portion of the particle size distribution at its small particle size end in accordance with 3.2.1 or 3.2.2.

4. Summary of Test Method

4.1 A screened and weighed sample of the carbon is placed in a special hardness pan with a number of stainless steel balls, then subjected to a combined rotating and tapping action for 30 min. At the end of this period, the amount of particle size degradation is determined by measuring the quantity of carbon, by weight, which is retained on a sieve whose openings are closest to one half the openings of the sieve that defines the minimum nominal particle size of the original sample.

5. Significance and Use

5.1 Several methods have been employed in the past for determining the resistance of activated carbons to particle size degradation under service conditions, including the ball-pan method, the stirring bar method, and the dust elutriation method. None of these has proved completely satisfactory for all applications, and all have been questioned by ASTM Committee D28 on Activated Carbon as tests for establishing degradation resistance. However, the ball-pan method has been used widely in the past and has a broad history in the activated carbon industry for measuring the property loosely described as "hardness." In this context the test is useful in establishing a measurable characteristic of a carbon. Conceding the fact that the test does not actually measure in-service resistance to degradation, it can be used to establish the comparability of lots ostensibly of the same grade of carbon.

TABLE 1 Hardness Test Sieve (HTS) Corresponding to Specification E11 Sieves Defining Small-End Nominal Particle Size (SNPS)

SNPS		HTS		SNPS		HTS	
Opening, mm	E11 Mesh	Opening, μm	E11 Mesh	Opening, μm	E11 Mesh	Opening, μm	E11 Mesh
5.6	3½	2800	7	850	20	425	40
4.75	4	2360	8	710	25	355	45
4.00	5	2000	10	600	30	300	50
3.35	6	1700	12	500	35	250	60
2.80	7	1400	14	425	40	212	70
2.36	8	1180	16	355	45	180	80
2.00	10	1000	18	300	50	150	100
1.70	12	850	20	250	60	125	120
1.40	14	710	25	212	70	106	140
1.18	16	600	30	180	80	90	170
1.00	18	500	35				

6. Apparatus and Materials

6.1 *Mechanical Sieve Shaker*, designed to produce from 140 to 160 taps and from 280 to 320 rotating motions per minute in a stack of standard Specification E11 sieves.³ Adjust the sieve shaker to accommodate the desired number of sieves, receiver pan, and sieve cover. Adjust the bottom stops to give a clearance of approximately 1.6 mm between the bottom plate and the sieves so that the sieves will be free to rotate. Fit the cover plate with a cork stopper which extends from 3.2 to 9.5 mm above the metal recess.

6.2 *Wire Cloth Sieves*, in accordance with Specification E11; six required, at least four of which bracket the expected nominal particle size distribution of the sample, and one of which, designated the hardness test sieve, has an opening as close as possible to one half the opening of the sieve that defines the smaller nominal particle size of the original sample. Table 1 lists the hardness test sieve corresponding to each minimum nominal sieve.

6.3 *Bottom Receiver Pan and Top Sieve Cover* (see 6.1).

6.4 *Hardness Test Pan*, having the dimensions of that in Fig. 1.

6.5 *Adjustable Interval Timer*, with a precision of at least ± 5 s, duration at least 600 s (10 min).

6.6 *Sample Splitter*, single-stage riffle type, in accordance with 30.5.2 of Practice E300.

6.7 *Balance*, with sensitivity and accuracy of at least 0.1 g.

6.8 *Soft Brass-Wire Brush*.⁴

6.9 *Steel Balls*, fifteen 12.7 ± 0.1 mm ($\frac{1}{2}$ in.) in diameter and fifteen 9.5 ± 0.1 mm ($\frac{3}{8}$ in.) in diameter.

7. Sampling

7.1 Guidance in sampling granular activated carbon is given in Practice E300.

³ The sole source of supply of the apparatus (the Tyler Ro-Tap Sieve Shaker, Model RX-29) known to the committee at this time is W.S. Tyler, Inc., Gastonia, NC. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁴ The sole source of supply of the apparatus (W.S. Tyler Model 1778-SB) known to the committee at this time is W.S. Tyler, Inc., Gastonia, NC. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

8. Calibration

8.1 Calibration of balances shall be maintained by standard laboratory methods. Sieves shall be calibrated at reasonable intervals in accordance with the procedure described in Specification E11.

9. Procedure

9.1 Determine the nominal particle size of the sample in accordance with Test Method D2862, and its moisture content in accordance with Test Methods D2867.

9.2 Obtain an additional representative sample of approximately 125 mL of the carbon in accordance with Practice E300.

9.3 Screen this sample to its nominal particle size distribution using Test Method D2862. Discard the fractions above the larger and below the smaller nominal particle size. Obtain at least 100 mL of material within the nominal mesh size range. Use additional material obtained as in 9.2, if necessary.

9.4 Measure out 100 mL of the screened sample into a tared, graduated cylinder in accordance with Test Method D2854, and weigh to the nearest 0.1 g.

9.5 Place the hardness pan (Fig. 1) on the standard bottom receiver pan. Pour the screened and weighed sample into the hardness pan and add the steel balls.

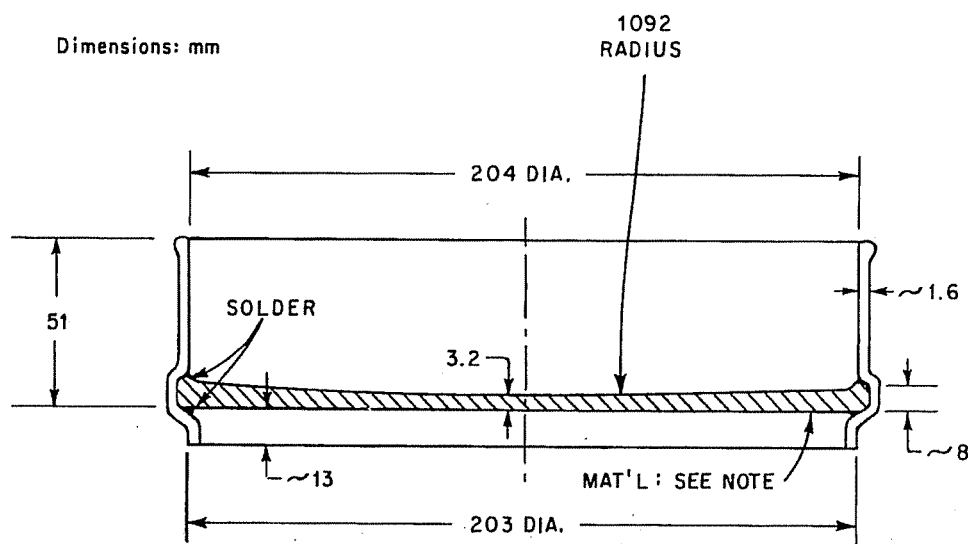
9.6 Complete the sieve stack by stacking five full-height sieves and the sieve cover on top of the hardness pan. The extra sieves, in this case, serve only to form a stack which fills the shaker, thus avoiding changes in tapping action and readjustment of the sieve stack retainer.

9.7 Place sieve stack in the sieve shaker and shake for 30 ± 0.5 min, with tapping hammer operating.

9.8 At the end of the shaking period, remove the sieve stack from the sieve shaker and remove the hardness pan from the sieve stack. Place the hardness test sieve on top of the receiving pan.

9.9 Remove the steel balls from the hardness pan and transfer sample to the hardness test sieve, brushing adhering particles into the sieve. Stack the five full-height sieves and sieve cover on top of the hardness test sieve and receiving pan, and replace the stack in the sieve shaker. Shake with the hammer operating for $10 \text{ min} \pm 10 \text{ s}$.

9.10 At the end of the shaking period, remove the sieve stack from the sieve shaker and transfer the remainder of the



NOTE 1—Material is plate, of one of the following alloys: (1) Cartridge brass, UNS C2600, half-hard temper, hardness 60 HRB or greater (see Specification B19); or (2) Aluminum bronze, UNS C6140, soft temper, hardness 140 HB or greater (see Specification B150/B150M).

FIG. 1 Pan for Ball-Pan Hardness Test

sample on the hardness test sieve to a tared weighing pan. Weigh to the nearest 0.1 g.

9.11 Sweep the pan catch into a tared weighing dish, and weigh to the nearest 0.1 g.

10. Calculation

10.1 Calculate the ball-pan hardness number from the equation

$$H = 100 B/A \quad (1)$$

where:

H = ball-pan hardness number,

B = weight of sample retained on hardness test sieve (see 9.10), g, and

A = weight of sample loaded onto hardness pan (see 9.4), g.

10.2 As a check on the accuracy of the test, calculate ball-pan hardness from the pan catch as follows:

$$H_2 = 100 (1 - C/A) \quad (2)$$

where:

C = pan catch from 9.11, g.

If H_2 differs from H by more than 2 %, one may assume that significant amounts of carbon are not accounted for, and the run must be rejected.

11. Report

11.1 Report the following information:

11.1.1 Name of the carbon supplier,

11.1.2 Grade designation of the sample,

11.1.3 Nominal particle size range and moisture content (as measured in 9.3),

11.1.4 Ball-pan hardness number,

11.1.5 Name of the agency and technician making the test,

11.1.6 Identification number and date of the test, and

11.1.7 Lot number from which the sample was taken.

12. Precision and Bias⁵

12.1 The precision of this test method is based on an interlaboratory study conducted in 2007. Each of nine laboratories tested four different materials. Every "test result" represents an individual determination. All laboratories submitted three replicate test results (from one operator) for each material.

12.1.1 *Repeatability*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the " r " value for that material; " r " is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

12.1.2 *Reproducibility*—Two test results shall be judged not equivalent if they differ by more than the " R " value for that material; " R " is the interval representing the difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

12.1.3 Any judgment in accordance with these two statements would have an approximate 95 % probability of being correct.

12.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

12.3 The precision statement was determined through statistical examination of 108 results, from nine laboratories, on four materials. These four carbons are described in Table 2.

12.4 As noted in the results table, the repeatability and reproducibility are dependent on the carbon type. To judge the equivalency of two test results, it is recommended to choose

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D28-1006.

TABLE 2 Hardness

Carbon	Average	Repeatability	Reproducibility	Repeatability	Reproducibility
	Standard	Standard	Standard	Limit	Limit
	Deviation	Deviation	Deviation		
	\bar{x}	sr	sR	r	R
Wood	62.37	4.16	16.76	11.66	46.92
45 sieve					
Lignite	80.20	1.96	4.46	5.49	12.48
50 sieve					
Coconut	99.36	0.18	0.51	0.51	1.44
16 sieve					
Bituminous	98.36	0.37	0.73	1.03	2.04
70 sieve					

the carbon closest in characteristics to the test carbon.

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