Designation: D 4164 – 03

Standard Test Method for Mechanically Tapped Packing Density of Formed Catalyst and Catalyst Carriers[1]

This standard is issued under the fixed designation D 4164; 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 the mechanically tapped density of formed catalyst and catalyst carriers. For the purpose of this test method, catalyst particles are defined as extrudates, spheres, or formed pellets of 0.8 to 4.8-mm (1⁄32 to 3⁄16-in.) nominal diameter.

1.2 *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]

D 3766 Terminology Relating to Catalysts and Catalysis

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E 456 Terminology Relating to Quality and Statistics

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—See Terminology D 3766.

4. Summary of Test Method

4.1 A preconditioned sample of formed catalyst or catalyst carrier is tapped in a graduated cylinder. The mechanically tapped packing density is determined from the known mass and tapped volume.

5. Significance and Use

5.1 This test method is to be used for measuring the mechanically tapped packing density of formed particles that will not break up during sampling, filling, or tapping of the measuring cylinder under test conditions.

6. Apparatus

6.1 *Graduated Cylinder*, capacity 250 mL with a base designed to accommodate the cylinder holder.

6.2 *Cylinder Holder*, weighing 1 lb (454 g).

6.3 *Tapping Device*, consisting of a base-plate with worm drive, reduction ratio 15:1, cam shaft speed of 250 r/min, tapping stroke travel 1⁄8 in. (3.2 mm).

6.4 *Four Digit Adjustable Counter*, which can be preset to deliver any number of taps between 1 and 9999.

6.5 *Desiccator*, with a desiccant grade molecular sieve such as No. 4A.

6.6 *Balance* having a sensitivity of 0.1 g.

6.7 *Drying Oven.*

7. Procedure

7.1 Heat an adequate sample(s) at 400 ± 15°C for not less than 3 h. Normally, this treatment can take place in air. However, in the case of materials that might react with air at elevated temperatures (such as prereduced catalysts) the heat treatment should take place in an inert atmosphere. After heating, cool the test sample(s) in a desiccator or other suitable container to eliminate the possibility of moisture adsorption prior to testing.

NOTE 1—These conditions may not be appropriate for all materials.

NOTE 2—Since many catalyst formulations are strong adsorbents, the use of No. 4A indicating (cobalt-treated) molecular sieves as a desiccating medium is suggested. The desiccant should be regenerated at 493 K (220°C) to 533 K (260°C), as required.

7.2 Pour between 240 and 250 mL of the test specimen carefully into the tared graduated cylinder using a funnel. To ensure proper level, rotate the funnel while pouring the test specimen. Weigh immediately to the nearest 0.1 g.

7.3 Preset the counter to 1000 taps.

7.4 Start the tapping device.

7.5 When tapping is completed, read the tapped volume, *V*, to the nearest 1 mL by estimating the average level of the catalyst surface in the cylinder.

[1] This test method is under the jurisdiction of ASTM Committee D32 on Catalysts and is the direct responsibility of Subcommittee D32.02 on Physical-Mechanical Properties.

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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.

8. Calculation

8.1 Calculate mechanically tapped packing density MTD as follows:

$$MTD = W/V \quad (1)$$

where:

W = mass of catalyst particles, g, and
V = volume occupied by particles in measuring cylinder, mL.

9. Precision and Bias [3]

9.1 *Test Program*—An interlaboratory study was conducted in which the named property was measured in two separate test materials in three separate laboratories. Practice E 691, modified for non-uniform data sets, was followed for the data reduction.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D32-1017.

9.2 *Precision*—Pairs of test results obtained by a procedure similar to that described in the study are expected to differ in absolute value by less than 2.772 S, where 2.772 S is the 95 % probability interval limit on the difference between two test results, and S is the appropriate estimate of standard deviation. Definitions and usage are given in Terminology E 456 and Practice E 177, respectively.

Test Result (consensus mean), kg/L	95 % Repeatability Interval (within laboratory), kg/L (% of mean)	95 % Reproducibility Interval (between laboratories), kg/L (% of mean)
0.6391	0.007 (1.02)	0.023 (3.54)
1.5333	0.005 (0.47)	0.055 (4.73)

9.3 *Bias*—The procedure in this test method has no bias because the value of mechanically tapped packing density can be defined only in terms of a test method.

10. Keywords

10.1 carriers; catalyst; mechanically tapped; packing density