Designation: D 4781 – 03

Standard Test Method for Mechanically Tapped Packing Density of Fine Catalyst Particles and Catalyst Carrier Particles[1]

This standard is issued under the fixed designation D 4781; 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 packing density of fine catalyst and catalyst carrier particles smaller than 0.8 mm in 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 dried fine catalyst or catalyst carrier particles 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 for measuring the mechanically tapped packing density of powders that are smaller than 0.8 mm in diameter, such as Fluidized Catalytic Cracking Catalysts (FCC).

6. Apparatus

6.1 *Graduated Cylinder*, capacity 100 mL.

6.2 *Holder*—A cylinder holder weighing 454 g (1 lb).

6.3 *Tapping Device*, consisting of a baseplate with worm drive, with specifications of 250 r/min camshaft speed, tapping stroke travel of 3.2 mm (⅛ in.).

6.4 *Counter*—A four-digit adjustable counter, which can be preset to deliver number of taps between 1 and 9999.

6.5 *Desiccator*, with a desiccant grade molecular sieve such as 4A.

6.6 *Balance* having a sensitivity of 0.1 g.

6.7 *Drying Oven.*

7. Procedure

7.1 Heat an adequate sample 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 temperature (such as prereduced catalysts) the heat treatment should take place in an inert atmosphere. After heating, cool the test sample 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 4A indicating (cobalt-treated) molecular sieves as a desiccating medium is recommended. The desiccant should be regenerated at 220 to 260°C, as required.

NOTE 3—Multiple samples can be pretreated but must be desiccated prior to analysis.

7.2 Carefully pour between 90 and 100 mL of the test specimen 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 tenth of a gram. The entire transfer time should be between 35 and 50 s.

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.

Current edition approved Oct. 1, 2003. Published October 2003. Originally approved in 1988. Last previous edition approved in 1999 as D 4781–99.

[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 the mechanically tapped packing density as follows:

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

where:

MTD = mechanically tapped packing density, g/mL,
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 four separate laboratories. Practice E 691, modified for non-uniform data sets, was followed for the data reduction. Analysis details are in the research report.

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), g/mL	95 % Repeatability Interval (Within Laboratory) g/mL (mean %)	95 % Reproducibility Interval (Between Laboratories) g/mL (mean %)
0.6195	0.005 g/mL (0.87)	0.060 (9.74)
0.9355	0.010 g/mL (1.07)	0.041 (4.41)

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

10. Keywords

10.1 catalyst; catalyst carrier; packing density

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D32-1026.