



Designation: D4512 – 22

# Standard Test Method for Vibrated Apparent Packing Density of Fine Catalyst and Catalyst Carrier Particles and Powder<sup>1</sup>

This standard is issued under the fixed designation D4512; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of the apparent packing density of fine catalyst and catalyst carrier powders smaller than 0.8 mm in diameter.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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*:<sup>2</sup>

- D3766 Terminology Relating to Catalysts and Catalysis
- E105 Guide for Probability Sampling of Materials
- E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E456 Terminology Relating to Quality and Statistics
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E1272 Specification for Laboratory Glass Graduated Cylinders

<sup>1</sup> 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 Aug. 1, 2022. Published August 2022. Originally approved in 1985. Last previous edition approved in 2018 as D4512 – 03(2018)<sup>ε1</sup>. DOI: 10.1520/D4512-22.

<sup>2</sup> 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. Terminology

3.1 *Definitions*—See Terminology D3766.

## 4. Significance and Use

4.1 This test method is for measuring the apparent packing density of catalyst or catalyst carrier powders that are smaller than 0.8 mm in diameter.

## 5. Apparatus

5.1 *Graduated Glass or Plastic Cylinders*, capacity 100 mL, feed and measuring, Class A or B per Specification E1272.

5.2 *Vibrator*,<sup>3</sup> conventional hand-held, with hard rubber or metal impactor.

5.3 *Feed Funnel*, plastic, glass, or metal as shown in Fig. 1.

5.4 *Ring Stand*, vibrator holder and clamps as shown in Figs. 2 and 3.

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

5.6 *Balance*, having sensitivity of 0.1 g.

5.7 *Drying Oven*.

## 6. Sample

6.1 Test sample shall be obtained from larger composites by riffing or splitting in accordance with subsection 5.12 of STP 447A<sup>4</sup> with the aim of obtaining a sample that represents the particle size distribution of the larger composite. Guide E105 can provide guidance on constructing a sampling plan with the representative sample size can be determined per Practice E122.

## 7. Procedure

7.1 Heat an adequate amount of sample(s) at  $400 \pm 15$  °C for not less than 3 h. Normally, this treatment can take place in

<sup>3</sup> The sole source of supply of the vibrator known to the committee at this time is Wahl Clipper Corp., Sterling, IL. They recommend their Model 4120 used at low speed to replace their Model 4180, which is discontinued. 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,<sup>1</sup> which you may attend.

<sup>4</sup> STP 447A, *Manual on Test Sieving Methods*, ASTM International, West Conshohocken, PA 19428.

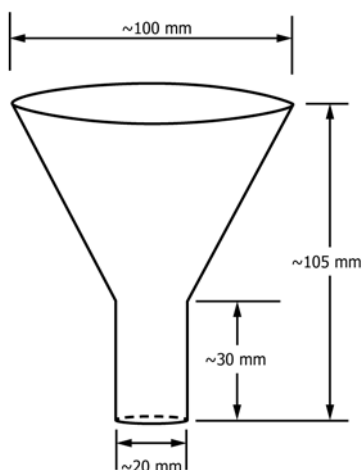


FIG. 1 Feed Funnel

air; however, in the case of materials that might react with air at elevated temperatures (such as, prereduced catalysts) the heat treatment shall 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 4A indicating (cobalt-treated) molecular sieve 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 Fill a feed glass cylinder with 100 mL of loosely packed, dried sample.

7.3 Turn on the vibrator and carefully add sample(s) to the tared, measuring cylinder through the feed funnel.

7.4 Transfer all of the sample to the measuring cylinder at a uniform rate not less than 2 mL or exceeding 3 mL/s. The entire transfer time shall be between 35 and 50 s.

7.5 After 60 additional seconds, turn off the vibrator. Read the vibrated volume,  $V$ , to the nearest milliliter by estimating the average level of the sample surface in the cylinder. Immediately weigh the sample and cylinder to the nearest tenth of a gram.

## 8. Calculation

8.1 Calculate the apparent packing density as follows:

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

where:

$APD$  = apparent packing density, g/mL,

$W$  = mass of sample particles, g, and

$V$  = volume occupied by sample particles in measuring cylinder, mL.

8.2 Apparent packing densities are typically reported to three significant figures.

8.3 The average of two or more runs is to be reported.

## 9. Precision and Bias<sup>5</sup>

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 E691, modified for nonuniform data sets, was followed for the data reduction.

9.2 *Precision*—Pairs of test results obtained by the procedure described in the method are expected to differ in absolute value by less than 2.772  $S$ , where 2.772  $S$  is the 95 % probability limit on the difference between two test results, and  $S$  is the appropriate estimate of standard deviation. Definitions and usage are given in Terminology E456 and Practice E177, respectively.

Test Result (Consensus)	95 % Repeatability Limit (Within Laboratory)	95 % Reproducibility Limit (Between Laboratories)
0.672 g/cm <sup>3</sup>	0.011 g/cm <sup>3</sup> (1.6 %)	0.090 g/cm <sup>3</sup> (13.4 %)
0.950 g/cm <sup>3</sup>	0.009 g/cm <sup>3</sup> (0.9 %)	0.062 g/cm <sup>3</sup> (6.6 %)

9.3 *Bias*—The procedure described is without bias since the property measured is defined in terms of the procedure.

## 10. Keywords

10.1 apparent density; catalyst; density

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D32-1005. Contact ASTM Customer Service at service@astm.org.

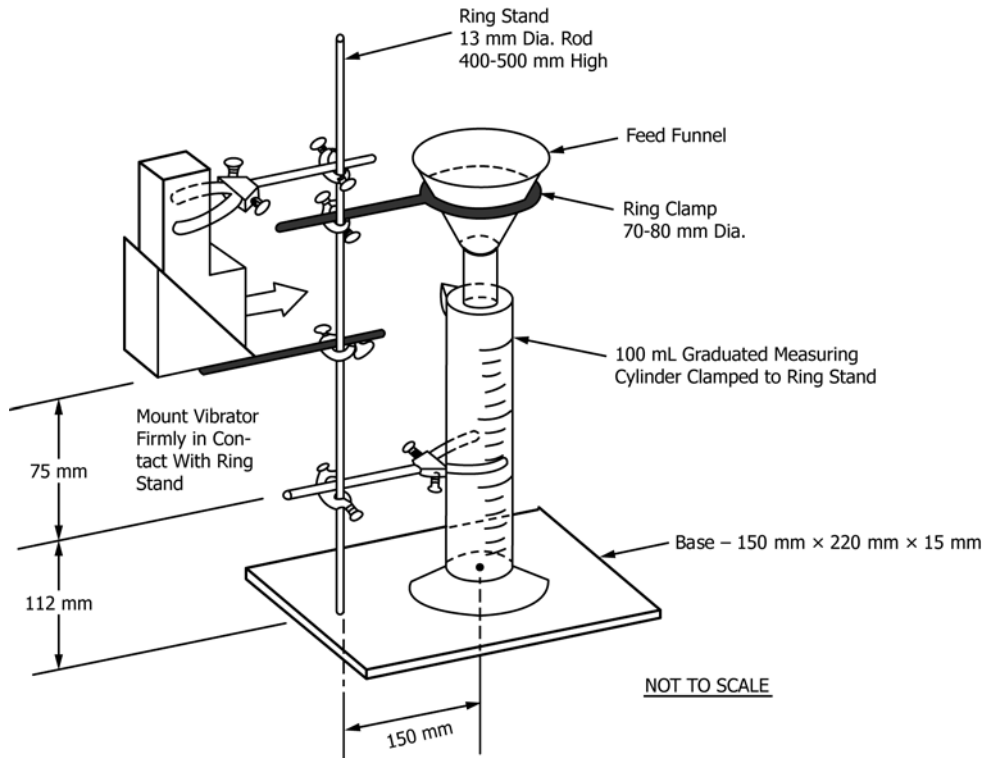


FIG. 2 Assembly of Apparatus

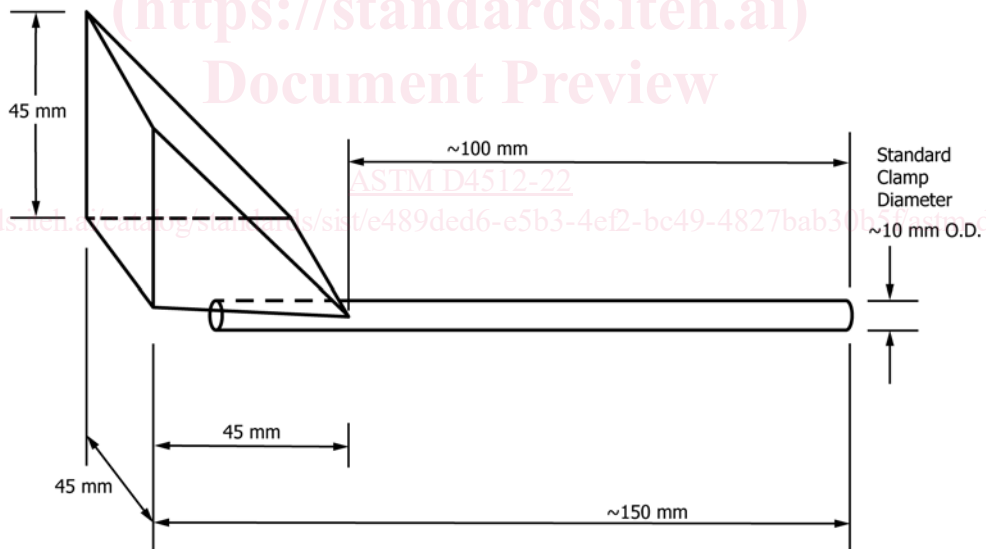


FIG. 3 Vibrator Holder