Designation: D4179 – 22

Standard Test Method for Single Pellet Crush Strength of Formed Catalysts and Catalyst Carriers[1]

This standard is issued under the fixed designation D4179; 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 determining the resistance of formed catalysts and catalyst carriers to compressive force and is applicable to regular catalyst shapes such as tablets and spheres. Other formed catalysts and catalyst carriers extrudates, granular materials, and other irregular shapes are specifically excluded.

1.2 This test method determines the average crush strength in the range from 0 to 50 lbf (0 to 220 N). Some materials may have crush strengths above 50 lbf (220N); the test method is applicable to these materials, but the precision of the test is not known.

1.3 *Units*—The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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:*[2]

E105 Guide for Probability Sampling of Materials

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *pellets, n*—any catalyst shape—tablets, spheres, or other similar configuration—that is not otherwise excluded from the scope of this test method.

3.1.2 *tablets, n*—tableted cylindrical catalyst particles, either solid or hollow core, with lengths that do not vary from the mean by more than ±10 %.

4. Summary of Test Method

4.1 Individual pellets taken from a representative sample are placed between two flat surfaces, subjected to a compressive load, and the force required to crush the pellet is measured. The procedure is replicated and the average of all measurements taken is determined.

5. Significance and Use

5.1 This test method is intended to provide information concerning the ability of a catalyst shape to retain physical integrity during use.

6. Apparatus

6.1 A suitable compression testing device is required, consisting of the following:

6.1.1 *Calibrated Gauge,* marked for direct reading of the force in pounds (newtons). Additionally, a suitable system (mechanical, hydraulic, or pneumatic) must be provided so that the rate of force application is both uniform and controllable within specified limits.

6.1.2 *Tool Steel Anvils,* between which the sample will be crushed. The faces of the tool steel anvils shall be smooth and free from recesses or ridges that would interfere with uniform contact along the major axis of the pellet. When testing tablets or spheres, the anvils may be of any convenient size or shape as long as their length and width are greater than the corresponding dimensions of the tablet or pellet being tested (see Fig. 1).

[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 Aug. 1, 2022. Published August 2022. Originally approved in 1982. Last previous edition approved in 2017 as D4179 – 11(2017). DOI: 10.1520/D4179-22.

[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.

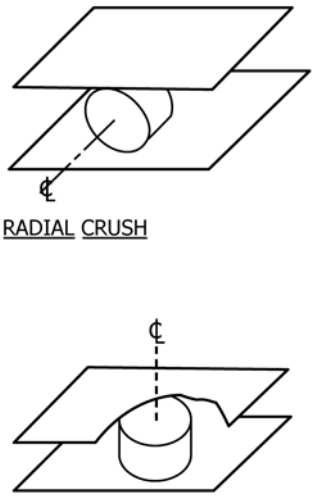

FIG. 1 Radial and Axial Crush

7. Sampling

7.1 A test sample of 50 to 200 individual pieces shall be obtained from larger composites by riffling or splitting in accordance with subsection 5.12 of STP 447A,[3] with the aim of obtaining a representative sample that represents shape and size distribution of the larger composite. The size of the sample shall depend on the precision required and the homogeneity of the material being tested. Guide E105 can provide guidance on constructing a sampling plan.

7.2 Pretreat the test 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 pre-reduced catalysts) the heat treatment should take place in an inert atmosphere. Care should be taken to ensure that the pretreatment does not alter the inherent strength or structure of the sample as evidenced by changes in surface area or phase. Any modifications to pre-treatment conditions should be noted in the report.

7.3 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—Since many catalyst formulations are strong adsorbents, the use of 4A indicating (cobalt-treated) molecular sieves as a desiccating medium is suggested. Regenerate the desiccant at 220 to 260 °C, as required.

8. Calibration and Standardization

8.1 Prior to use, set the test apparatus to zero and calibrate with any commercially available force gauge with marked graduations of no more than ½ lbf (2 N) and having accuracy traceable to the National Institute of Standards and Technology, or other similar authority.

[3] STP 447A, *Manual on Test Sieving Methods*, ASTM International, West Conshohocken, PA 19428.

9. Procedure

9.1 Remove from the desiccator only that number of pellets that can be tested within a 10 min period. (**Warning—**Ensure that moisture pick-up in the 10 min period will not significantly affect the pellet crush strength.)

9.2 Place a single catalyst pellet between the anvils of the compression testing device. Orient each pellet in the same direction before crushing. For those pellets capable of being tested in different orientations, report the one used. Fig. 1 shows pellets in radial and axial orientations. Use tweezers, forceps, or other suitable device or procedure to prevent the transfer of moisture from the operator's hands to the piece being tested.

9.3 Piston is to be advanced at a constant rate of about 0.4 in./min (1 cm/min) such that the applied force is in the range of 1 to 10 lbf/s (4.4 to 44 N/s) until the pellet crushes or collapses. Compression of surface irregularities or limited fracturing of a pellet followed by continued resistance to increasing load are not to be used as criteria for determining the endpoint of this test.

9.4 Read and record, to the nearest one-half graduation, the force indicated on the calibrated dial of the apparatus at the instant of collapse.

9.5 Separate the anvils and remove all residue with a soft cloth or brush. Ensure that the faces of the anvils are free from adhering particles.

9.6 Repeat steps 9.2 through 9.5 until all pellets in the sample have been crushed. Record the crush strength for each pellet tested.

9.7 Steps 9.3 – 9.5 can be automated.

10. Calculation

10.1 Calculate the average crush strength ($\bar{x}$), retaining one more decimal place than the recorded values, as follows:

$$\bar{X}\ \text{lbf}\ (N) = \left(\sum X\right)/(n) \tag{1}$$

where:

$\sum X$ = the sum of all observed crush strengths, and
n = the number of pellets crushed.

10.2 Calculate the standard deviation of the n readings to three significant digits as follows:

$$S = \sqrt{\frac{\sum \left(X - \bar{X}\right)^2}{\text{n} - 1}}\ \text{lbf}\ (N) \tag{2}$$

where:

S = standard deviation of the individual strength values, and
$\sum(X - \bar{X})^2$ = sum of the squares of the deviations of each recorded reading from the average strength.

NOTE 2—Many calculators are programmed to perform these operations and to report average and standard deviation directly. It is important to verify that the program chosen uses the $n - 1$ denominator rather than n in calculating standard deviation.

11. Report

11.1 Report the average crush strength to one more decimal place than the recorded data on the individual strengths. For