Designation: D 4179 – 01

Standard Test Method for Single Pellet Crush Strength of Formed Catalyst Shapes[1]

This standard is issued under the fixed designation D 4179; 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 to compressive force and is applicable to regular catalyst shapes such as tablets and spheres. 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).[2]

1.3 The values stated in inch-pound units are to be regarded as the standard. The values in parentheses for information only.

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

2. Referenced Documents

2.1 *ASTM Standards:*

E 177 Practice for Use of the Terms Precision and Bias in ASTM Methods[3]

E 456 Terminology Relating to Quality and Statistics[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System[2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

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

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 Gage*, 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).

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 STP 447A,[4] (paragraph 5.12) 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.

7.2 Heat 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 prereduced catalysts) the heat treatment should take place in an inert atmosphere.

7.3 After heating, cool the test sample(s) in a desiccator or other suitable container to eliminate the possibility of moisture

[1] This test method is under the jurisdiction of ASTM Committee D32 on Catalysts and is the direct responsibility of Subcommittee D 32.02 on Physical Mechanical Properties.

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[2] *Annual Book of ASTM Standards*, Vol 14.04.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from ASTM International Headquarters, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428–2959, STP 447A, *Manual on Test Sieving Methods*.

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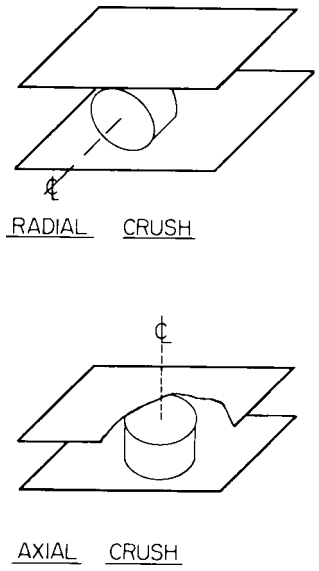

FIG. 1 Radial and Axial Crush

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 gage 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.

9. Procedure

9.1 Remove from the desiccator only that number of pellets that can be tested within a 10-min period.

NOTE 2—**Caution:** Take care to assure 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 Apply increasing force at a uniform rate 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 end-point 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 9.6 until all pellets in the sample have been crushed. Record the crush strength for each pellet tested.

10. Calculation

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

$$\bar{X} \text{ lbf } (N) = (\Sigma X)/(n) \quad (1)$$

where:
ΣX = 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 3 significant digits as follows:

$$S = \sqrt{\frac{\Sigma (X - \bar{X})^2}{n - 1}} \text{ lbf } (N) \quad (2)$$

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

NOTE 3—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 pellets capable of being tested in different orientations, the one used should be reported.

11.2 Report the "80 % spread" (that is, the range within which 80 % of the individual pellet strengths are expected to fall, assuming that individual pellets form a "normal" distribution). Calculate as follows:

$$80 \text{ \% spread} = \bar{X} \pm 1.28S \quad (3)$$

11.3 Report the "95 % Reliability" of the average reported in 11.1. This is the uncertainty inherent in the reported average, expressed as the range within which 95 % of the averages of an infinite number of test samples of n pellets would be expected to fall were they to be similarly drawn from the same lot and tested. Calculate as follows:

$$95 \text{ \% reliability} = \bar{X} \pm 1.96\, S/\sqrt{n} \quad (4)$$

11.4 Report the applied rate of force increase, if available.

12. Precision and Bias [5]

12.1 *Test Program*—An interlaboratory study was conducted in which the named property was measured in two separate test materials in six separate laboratories. Practice E 691, modified for nonuniform data sets, was followed for the data reduction. Analysis details are in the research report.

12.2 *Precision*—Pairs of test results obtained by a procedure similar to that described in the study are expected to differ

[5] Supporting data are available from ASTM International Headquarters. Request RR: D32–1004 and RR: D32–1024.

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) lbf	95 % Repeatability Interval (Within Laboratory) lbf (% of mean)	95 % Reproducibility Interval (Between Laboratories) lbf (% of mean)
20.19 (Spheres)	1.22 (6.03)	1.76 (8.74)
16.50 (Tablets)	1.00 (6.03)	2.77 (16.8)

12.3 *Bias*—This method is without known bias.

13. Keywords

13.1 catalyst; crush strength; single pellet

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