Designation: D6175 – 03 (Reapproved 2008)

Standard Test Method for Radial Crush Strength of Extruded Catalyst and Catalyst Carrier Particles[1]

This standard is issued under the fixed designation D6175; 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 and is suitable for determining the resistance of extruded catalysts and catalyst carriers to compressive force from the side.

1.2 This test method was developed using extruded catalyst and catalyst carriers from 1⁄16 to 1⁄8 in. in diameter (0.159 to 0.318 cm) and limited to pieces with a length to diameter ratio greater than or equal to 1:1. This test method may be applicable to other diameters.

1.3 This test method is suitable for the determination of mean crush strength per millimetre in the range of 0 to 15 lbf/mm (0 to 65 N/mm).

1.4 The values stated in pounds lbf/mm units are to be regarded as the standard. The values given in parentheses are provided for information only.

1.5 *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 to determine the applicability of regulatory limitations before use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3766 Terminology Relating to Catalysts and Catalysis

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*—See Terminology D3766.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *extruded catalyst particles*—cylindrical particles with uniform cross sections, either solid, hollow core, or multilobed, formed by extrusion.

4. Summary of Test Method

4.1 Individual extrudates taken from a representative sample are calcined, measured in length, placed between two flat surfaces, and subjected to a compressive force. The force required to crush the extrudate is measured. The procedure is replicated, the force per millimetre calculated, and the average of all quotients determined.

5. Significance and Use

5.1 This test method is intended to provide information on the ability of an extruded catalyst to retain physical integrity during use.

6. Apparatus

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

6.1.1 *Calibrated Pressure or Force Gage*, marked for direct reading of the force in pounds force (Newtons) with a range about two times the expected average force reading. A suitable system (mechanical, hydraulic, or pneumatic) must be provided so that the rate of force applied is both uniform and controllable within specified limits (see 9.4).

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 hollows or ridges that would interfere with uniform contact along the length of the extrudate. The faces shall be parallel to each along their entire length of travel. The faces of both anvils must be longer in one dimension than the length of the sample pieces to be crushed.

6.2 A device for determining length, reading in millimetres, and of suitable accuracy to measure to the nearest tenth.

7. Sampling

7.1 A test sample of 50 to 200 individual particles shall be obtained from larger composites by riffling or splitting according to STP 447A,[3] with the aim of obtaining a representative

[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 April 1, 2008. Published April 2008. Originally approved in 1998. Last previous edition approved in 2003 as D6175–03. DOI: 10.1520/D6175-03R08.

[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.

[3] STP 447A, *Manual on Test Sieving Methods*, Section 5.12, ASTM International, W. Conshohocken, PA. This title is out of print.

sample that represents both the shape and size of the larger composite. The amount of the sample shall depend on the precision required and the homogeneity of the material being tested. All of the individual particles sampled that have a length to diameter ratio greater than or equal to one shall be tested.

7.2 Heat the test sample(s) at 400 ± 15°C for not less than 3 h.

NOTE 1—Moisture pick-up by extrudates is often rapid and the measured crush strength may be affected.

7.3 After heating, cool the test sample(s) in a desiccator or other suitable container to prevent the adsorption of moisture before testing.

NOTE 2—If the catalyst may be damaged at 400°C, a lower temperature can be used so long as it is specified with the result. Normally, this treatment can take place in air. However, for materials that might react with air at elevated temperatures (such as prereduced catalysts), the heat treatment should take place in an inert atmosphere.

NOTE 3—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 Before use, the test apparatus should be set to zero and calibrated 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 for Standards and Technology or other similar authority.

9. Procedure

9.1 Remove from the desiccator only that number of extrudates that can be tested within a 10-min period. To hold the dry extrudates, the use of an upright fritted disk funnel equipped with a dry, upward-flowing, inert gas purge is suggested.

NOTE 4—Precaution must be taken to ensure that moisture pick-up in the 10-min period will not significantly affect the extrudate crush strength.

9.2 Measure and record the length of an extrudate to the nearest tenth of a millimetre. 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 Then place the measured extrudate that has a length to diameter ratio greater than or equal to one between the anvils of the compression testing device. The extrudates shall be flat against the face of the anvil and be crushed radially (see Fig. 1).

9.4 Apply increasing force at a uniform rate in the range of 1 to 5 lbf/s (4.4 to 22 N/s) until the extrudate crushes or collapses. Compression of any surface irregularities or limited fracturing of the extrudate followed by continued resistance to increasing load are not be used as criteria for determining the endpoint of this test.

9.5 Read and record, to the nearest one-half graduation or first decimal place, the force indicated on the calibrated meter of the apparatus at the instant of collapse. A pressure holding or recording device coupled to the moving anvil is suggested to indicate the pressure at the time of collapse.

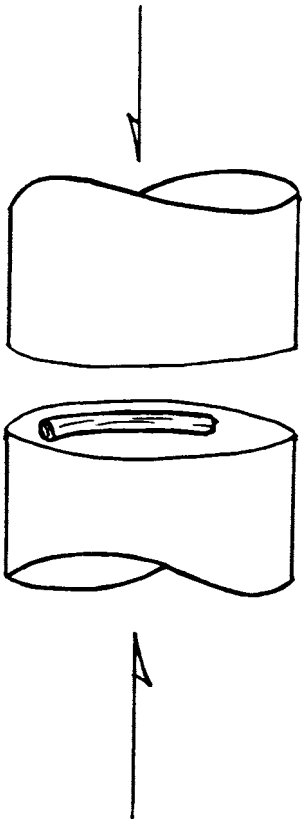

FIG. 1 Placement of Extrudate Between Anvils

9.6 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.

9.7 Repeat 9.2-9.6 until all extrudates with a length to diameter ratio greater than or equal to one in the sample have been crushed.

10. Calculation

10.1 Calculate the crush strength per millimetre (x) for each extrudate, retaining the results to the nearest tenth unit, as follows:

$$x = \frac{F}{L} \tag{1}$$

where:

x = the crush strength for one extrudate per millimetre, lbf/mm (N/mm),
F = the force necessary to crush the extrudate, lbf (N), and
L = the length of the extrudate along its cylindrical axis, mm.

10.2 Calculate the mean crush strength per millimeter (X) as follows, retaining the results to the nearest tenth unit:

$$X = \Sigma x / n \tag{2}$$

where:

X = mean crush strength millimetre, lbf/mm (N/mm),
Σx = the sum of the observed crush strengths per millimetre, and
n = the number of extrudates crushed.

10.3 Calculate the standard deviation of the n readings (to three significant digits):

$$S = \sqrt{\frac{\sum (X - \bar{X})^2}{n - 1}} \text{ lbf } (N) \quad (3)$$

where:

S = standard deviation of the individual strength in lbf/mm (N/mm).

$\Sigma(X - \bar{X})^2$ = the sum of the squares of the deviations of each individual strength per millimetre from the average strength per millimetre.

NOTE 5—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 mean crush strength per millimetre (X) to the nearest tenth unit and the standard deviation (S).

11.2 Record and report the number of particles measured.

11.3 If applicable, report the method used to obtain the analysis sample from the parent sample.

12. Precision and Bias

12.1 *Test Program*—An interlaboratory study was conducted in which the named property was measured in two separate test materials in six and seven laboratories. Practice E691, modified for non-uniform data sets, was followed for the data reduction. See Table 1.

12.2 *Precision*—Pairs of test results obtained by the procedure described in the test method are expected to differ in absolute value by less than 2.772 S, where 2.772 S is the 95 % probability interval 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.

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

13. Keywords

13.1 catalyst; crush strength; extrudate; radial

TABLE 1 Test Program Results

Test Results (Consensus Means)	95 % Repeatability Interval (Within Laboratory)	95 % Reproducibility Interval (Between Laboratories)
4.15 lbf/mm	0.16 lbf/mm (3.90 % of mean)	0.834 lbf/mm (20.5 % of mean)
3.95 lbf/mm	0.17 lbf/mm (4.25 % of mean)	0.641 lbf/mm (16.8 % of mean)