Designation: D7084 – 04 (Reapproved 2009)

Standard Test Method for Determination of Bulk Crush Strength of Catalysts and Catalyst Carriers[1]

This standard is issued under the fixed designation D7084; 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 bulk crush strength of a bed of formed catalyst particles 1⁄32 to 3⁄16 in. (0.8 to 4.8 mm) in diameter and is intended to provide information concerning the ability of the catalyst material to maintain physical integrity.

1.2 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.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3766 Terminology Relating to Catalysts and Catalysis

D4180 Test Method for Vibratory Packing Density of Formed Catalyst Particles and Catalyst Carriers

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:*

3.1.1 See also Terminology D3766.

3.1.2 *bulk crush strength*—pressure that generates 1 % fines for a sample contained in a cylindrical sample holder and crushed with a piston.

3.1.3 *generated fines*—particle size after crushing that passes through a sieve one-half of the diameter of the catalyst pellet.

4. Summary of Test Method

4.1 A representative sample is placed in a cylindrical sample holder, which is fitted with a piston. The piston is allowed to compress the catalyst at a known pressure. The percent of generated fines is determined by sieving.

5. Significance and Use

5.1 This test method is a means of determining the crushing strength of a catalyst in a bed. Techniques to measure the crushing strength of formed catalyst particles is limited to crushing of individual particles, which may not be related to how the catalyst will crush in a reactor or bed. For some catalysts, such as granules, this technique may be the only viable method for obtaining crushing strength. The production of fines in a reactor is not desired because of the potential of bed compaction and the pressure buildup in the reactor.

6. Apparatus

6.1 *Hydraulic Press*, capable of 3200 lb (1450 kg) loading, including a force gage. Maximum load capacity of the press should match with the accuracy measuring the applied force.

NOTE 1—Lower maximum load may be acceptable for testing less strong materials.

6.2 *U.S. Standard Sieves*, set (Tyler Equivalent).

6.3 *Test Cell*, (Fig. 1).

NOTE 2—A top loading cell can be used, but reproducibility of this test is a function of the volume being crushed and therefore the constant volume cell, as shown in Fig. 1, is recommended. A smaller *l/d* ratio for the cell will alter the results, and for certain applications, it may be more desirable.[3,4]

6.4 *Drying Oven*.

6.5 *Balance*, having a sensitivity of 0.1 g.

[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.

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[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] Bradley, S. A., Pitzer, E., and Koves, W. J., "Bulk Crush Testing of Catalysts," *Characterization and Catalyst Development*, ACS Symposium Series 411, Bradley, S. A., Gattuso, M. J., and Bertolacini, R. J., Eds., 1989, pp. 398-406.

[4] Adams, A. R., Sartor, A. F., and Welsh, J. G., "Problems in Standardizing Catalyst Tests," *Chemical Engineering Progress*, Vol 71, 1975, pp. 35-37.

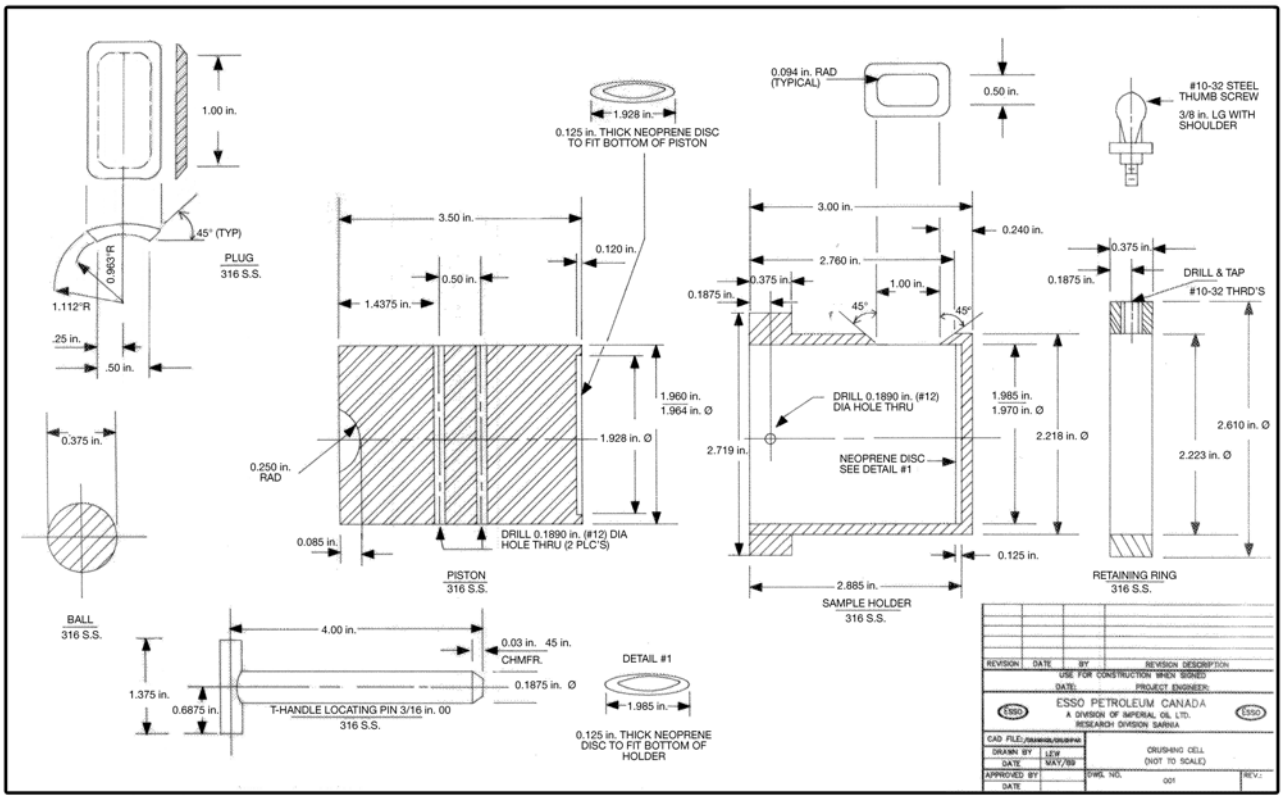

FIG. 1 Test Cell

6.6 *Glass Cylinders*, with 250-mL feed capacity. Measure in accordance with Test Method D4180.

6.7 *Vibrator*, conventional, handheld, with hard rubber or metal impactor, in accordance with Test Method D4180.

6.8 *Feed Funnel*.

6.9 *Desiccator*, with grade molecular sieve, such as 4A.

7. Procedure

7.1 Determine the bulk density in accordance with Test Method D4180.

7.2 Weigh 100 mL of the sample and dry at 673 ± 15 K (400°C) for 3 h. Cool the sample in a desiccator using freshly regenerated 4A molecular sieves as a desiccant.

NOTE 3—These conditions may not be appropriate for all materials.

NOTE 4—Since many catalyst formulations are strong adsorbents, the use of No. 4A indicating (cobalt treated) molecular sieve as a desiccating medium is suggested. The desiccant should be regenerated at 493 to 523K (220 to 260°C) as required.

7.3 Weigh the sample to determine the loss during heating. Calculate the dry bulk density by the following equation:

$$DBD = BD\,(1 - (OW - DW)/OW) \quad (1)$$

where:

DBD = dry bulk density (see Test Method D4180),
BD = bulk density,
OW = weight of original sample, and
DW = weight of dried sample.

7.4 Calculate the weight of dried sample required to fill a volume of 49 cm^3, and then calculate the original weight of this sample.

7.5 Weigh sufficient sample for nine crushing operations. Sieve the sample using a sieve that is one-half the diameter of the catalyst pellet. If 10 % of the material passes through the screen, use a smaller sieve size. Dry the sample at 673 K for 3 h; do not exceed a bed height of 1 in. Cool the sample in a desiccator.

7.6 Weigh a sample required to fill a volume of 49 cm^3, as determined in 7.4. Keep the remaining sample in the desiccator.

7.7 Fill the cell with the 49-cm^3 dried sample. Tap the cell while filling it. Place the entire catalyst sample into the cell. Place and center the cell into the hydraulic press.

7.8 Apply a pressure to the cell at a rate of 50 lb/$in.^2$/s (35 kPa/s), and read the pressure. Hold the prescribed pressure for 30 s, and then slowly release the pressure.

7.9 Remove the cell from the press, and then remove the piston from the cell. Brush all of the particles from the end of the piston into the sieve in 7.5. Pour the contents of the cell onto the sieve, and brush all particles from the cell into the sieve.

7.10 After sieving the fines for 60 s into a weighing pan, weigh the catalyst in the pan. Perform sieving so as not to produce additional fines.

7.11 Crush strengths are determined in triplicate for at least two different pressures that will produce both less than and more than 1 weight percent fines. Typical loads are between 150 and 500 lb/$in.^2$ (1 and 3.5 MPa) for larger formed materials and 15 and 50 lb/$in.^2$ (0.1 and 0.35 MPa) for granules.

7.12 Use a fresh charge of catalyst for each loading in order to prevent bias of damaged catalyst or being left with stronger pellets.

7.13 Weight percent fines is determined by:

$$F = PW/DW \times 100 \quad (2)$$

where:

F = weight percent fines,
PW = weight of fines in pan after sieving, and
DW = dry weight of sample put into cell.

7.14 Plot crushing pressure versus weight percent fines. Draw a straight line having the best correlation coefficient. Report the crush strength for 1 % fines generation. Crushing strength is the applied force divided by the cross-sectional area of the interior of the cell. For the recommended cell the area is 3.14 in.^{2} (20.26 cm^2).

8. Report

8.1 Report the following information:

8.1.1 Plot of the crushing pressure versus the weight percent fines,

8.1.2 Correlation coefficient,

8.1.3 Interpolated crush pressure to produce 1 % fines,

8.1.4 Sieve size used, and

8.1.5 Detail of any changes in the loading cell.

9. Precision and Bias

9.1 *Test Program*—An interlaboratory study was conducted in which the named property was measured in three separate test materials in three separate laboratories. The three catalysts conducted in this study were of different geometries, densities and sizes. Practice E691 was followed for the data reduction. Analysis details are in the research report.[5]

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 Practice E177 and Terminology E456.

Test Result (Consensus Mean)	95 % Repeatability Interval (Within Laboratory)	95 % Reproducibility Interval (Between Laboratories)
189.3 psi (1.30 MPa)	26.02 psi (0.18 MPa) (13.75 % of mean)	32.37 psi (0.22 MPa) (17.11 % of mean)
198.5 psi (1.36 MPa)	25.60 psi (0.18 MPa) (12.89 % of mean)	48.14 psi (0.33 MPa) (24.25 % of mean)
186.5 psi (1.28 MPa)	34.41 psi (0.24 MPa) (18.45 % of mean)	78.83 psi (0.54 MPa) (42.26 % of mean)

9.3 *Bias*—The procedure in this test method has no known bias because the value is defined only in terms of this test method.

10. Keywords

10.1 bulk crush strength; bulk density; formed catalyst particles

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D32-1040.