

AUTOMATED CRUSH STRENGTH OF CATALYSTS OR MOLECULAR SIEVES

UOP Method 914-92

SCOPE

This method is for determining the crush strength of catalysts and molecular sieves using an Erweka tablet hardness testing instrument. The method applies to extrudates or spheres, herein referred to as particles, generally having a nominal diameter of 1.6 mm (1/16 inch) or larger. The detection range is from 250 Newtons (N) (56 lb_f) down to 5 N (1 lb_f).

OUTLINE OF METHOD

Thirty representative particles from a sample are individually subjected to an increasing linear force between a fixed and a motor-driven anvil. Standard flat anvils are used for spherical particles while specially fabricated semicircular ones are used for extrudates (see Note 1). At the point of crush, the motor is stopped automatically and the force required to crush the particle is displayed on a digital readout and printer. The average crush strength is calculated from the arithmetic average of the force required to crush 30 particles. The percent particles having a crush strength below 22 N (5 lb_f) can be reported as well as the maximum and minimum crush strength for individual particles within the set. In addition, the crush strength data from the 30 particles may be graphically displayed as a histogram.

APPARATUS

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Anvils, 5.0-mm radius, semicircular, 316/316L stainless steel, Fig. 1, set of 2, UOP

Brush, round, camel's hair, Fisher Scientific, Cat. No. 03-670

Calibration brackets, Erweka Instrument, Cat. No. 1058

Calibration weights, Erweka Instrument, Cat. No. 1058/A

Computer, personal, capable of running Lotus 1-2-3, or similar, software to develop a histogram (optional)

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

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UOP Methods are available through ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken PA 19428-2959, United States. The Methods may be obtained through the ASTM website, www.astm.org, or by contacting Customer Service at service@astm.org, 610.832.9555 FAX, or 610.832.9585 PHONE.

Forceps, blunt-pointed, straight, 127-mm length, Fisher Scientific, Cat. No. 08-890

Goggles, monogoggle, VPC, Fisher Scientific, Cat. No. 17-283-2

Level, calibrated, circular, precision, McMaster Carr, Cat. No. 2198A85

Tablet hardness tester, Erweka Model TBH-28-DR, with printer, Erweka Instrument, Cat. No. 1050-1052. Other equipment is not acceptable for this method.

Tray, utility, stainless steel 420- x 250- x 64-mm, Fisher Scientific, Cat. No. 13-361C

Vacuum pick-up system, electric, McMaster Carr, Cat. No. 5670A1 (optional)

REAGENTS AND MATERIAL

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Printer paper, Erweka TBH-28, Cat. No. 1000

Tape, 810 Scotch Brand, 3/4-inch width, Boise-Cascade, Cat. No. A8-1296-3/4

Tape, Scotch Brand, double-coated, 3/4-inch x 36-yd, Boise-Cascade, Cat. No. A8-665-3/4

PROCEDURE

Calibration

The Erweka instrument manual must be thoroughly understood before operation or calibration. The instrument is to be calibrated per Erweka instructions.

It is suggested that the user select suitable spherical and extrudate reference catalysts or molecular sieves for weekly quality assurance/quality control (QA/QC) evaluation. Sufficient reference material should be obtained to last for at least 6 months. The reference should be well-riffled and stored in sealed containers to reduce moisture absorption. The reference data obtained can be used to develop a control chart that is valuable in detecting analytical variations. Recalibrate the instrument as indicated by the control chart, or if QA/QC is not used, at least once per month.

Instrument Setup

1. Attach the circular level to the crushing mechanism using double-coated tape (see Fig. 2).
2. Level the instrument crushing mechanism using any convenient shim material under the rubber feet of the instrument.
 - The leveling operation reduces the tendency of spherical particles to roll on the platform.
3. Apply a strip of 810 Scotch tape approximately four layers thick (see Fig. 2) down the center of the platform between the two anvils.
 - The tape serves as an aid in maintaining a sphere at the centerline of the anvil.
 - The tape is not required when extrudates are being analyzed.

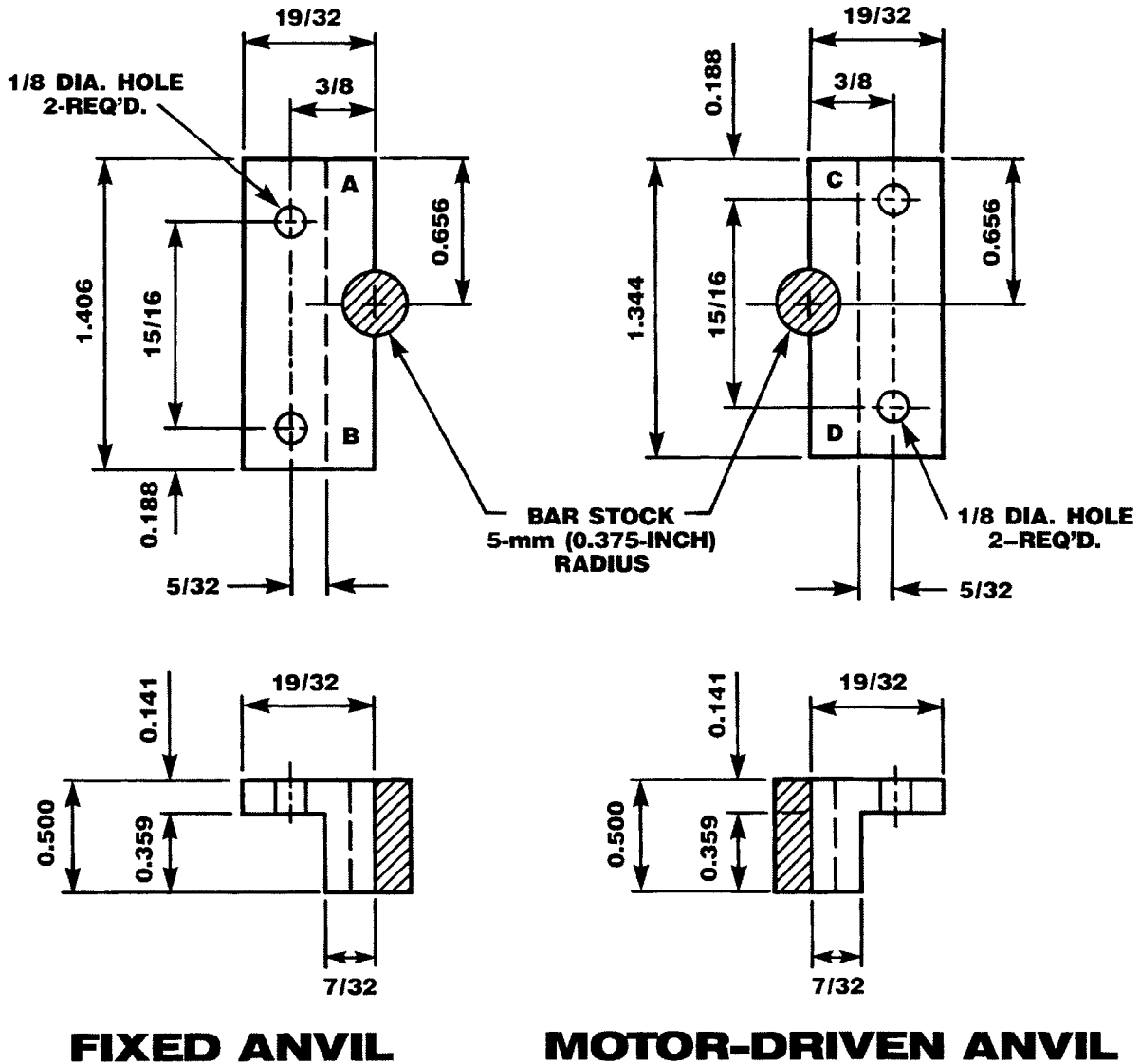


Figure 1
Semicircular Anvils

ALL DIMENSIONS IN INCHES

TOLERANCE ± 0.005 - DECIMAL

$\pm 1/32$ - FRACTIONAL

RADIUS IS MILLED TO FIT DIAMETER OF BAR STOCK.

BAR STOCK IS TACK WELDED IN PLACE, FLUSH AT BOTTOM.

WHEN RADIUS IS MILLED INTO ANVIL ENDS "A" AND "C" MUST BE FLUSH WITH EACH OTHER.

MATERIAL TO BE TYPE 316/316L STAINLESS STEEL

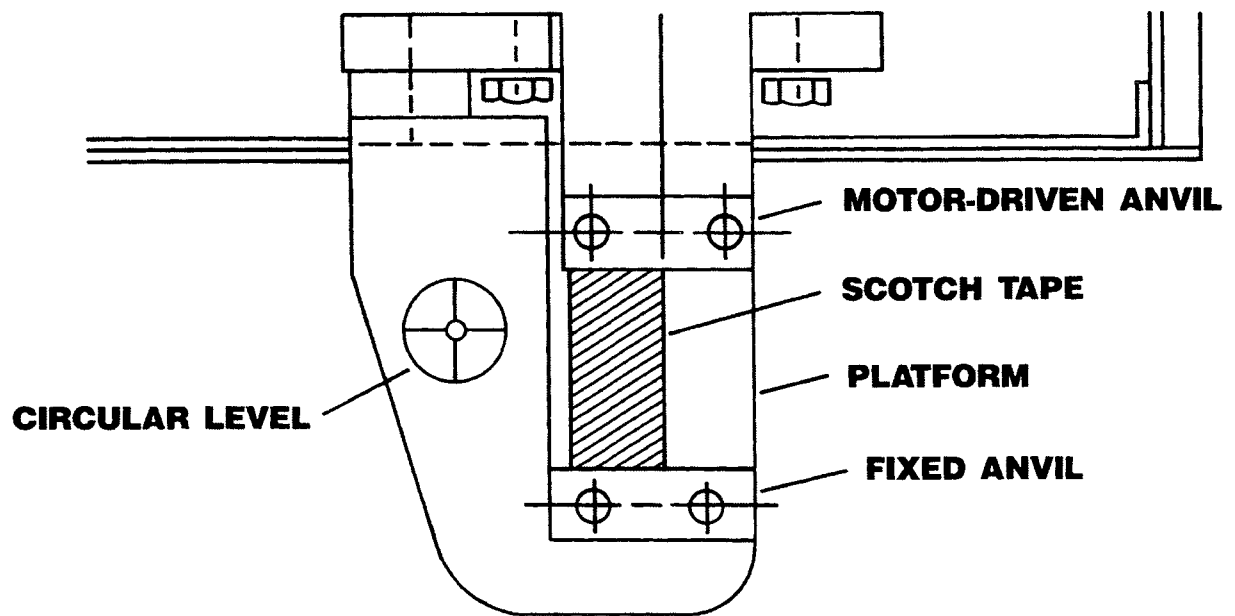


Figure 2
Crushing Mechanism

Spherical Particles

1. Obtain 30 pieces of catalyst or molecular sieves that are representative of the sample.
 - The instrument is equipped with flat anvils for the analysis of spherical particles.
2. Set the piece diameter to 5 mm for all particle sizes up to 3.2-mm diameter with the electronic coding switch.
3. Press the printer button.
 - The red light will illuminate.
4. Set the units of force to “N” with the “KP-N-SC” button.
5. Press the button until the illuminated dot is in the “N” position.
6. Set the illuminated dot to “AUTO” with the “Auto-O-Man” button.
7. Position a particle at the center of and against the fixed anvil using the blunt forceps or the vacuum pick-up system.
 - The particle must remain centered and not be allowed to roll from this position.
 - Molecular sieves or other very hygroscopic materials should be blanketed with dry nitrogen until they are placed in the instrument for crushing.
8. Press “START” to begin movement of the motor-driven anvil.
 - After the crush, the moveable anvil automatically returns to the preset position.
9. Brush the crushed material from the anvils into the utility tray with the camel’s hair brush.
10. Insert the next particle immediately.
 - After a pause of 3 seconds the unit starts the next test automatically.
 - If the next particle cannot be inserted within the 3 second time, wait until the unit has returned to the starting position and press “START” again to continue testing.
 - If the analyst finds the instrument recycle speed too fast, the piece diameter setting can be increased until a comfortable interval is determined. The piece diameter setting can then be decreased as the analyst establishes a routine.
11. Repeat Steps 9 and 10 until all 30 pieces have been crushed.
12. Press “START” to stop the test after the 30th particle has been crushed.
13. Set the illuminated dot to “O” with the “Auto-O-Man” button for the report printout.

Extruded Particles

Replace the 2 flat anvils with 5.0-mm semicircular anvils as follows:

1. Remove the motor-driven flat anvil by loosening the two screws that hold it in place.
2. Position the shorter semicircular motor-driven anvil (see Fig. 1) in place and secure it with the screws.
3. Install, the longer semicircular fixed anvil in the same way.

4. Obtain 30 pieces of catalyst or molecular sieves that are representative of the sample.
 - Efforts should be made to randomize the pieces chosen for testing.
5. Set the piece diameter to 25 mm for particles up to 3.2-mm diameter with the electronic coding switch.
6. Place the extrudate horizontally on the platform, parallel to and against the face of the fixed anvil. Center the piece at the center of the anvil.
7. Continue the analysis as described in Steps 3 through 12 under *Spherical Particles*.
 - Molecular sieves or other very hygroscopic materials should be blanketed with dry nitrogen until they are placed in the instrument for crushing.
 - A simple fracture of the extrudate rather than a true crush can usually be detected as a low measurement, not typical of values obtained for other pieces in the sample set. Such a measurement is to be rejected and an additional piece run. In such cases, manual calculation of average properties will be required since the automated calculation program of the Erweka instrument does not provide for rejecting values.

CALCULATIONS

The calculation of average crush strength is pre-programmed into the microprocessor of the Erweka Hardness Tester and the result is automatically printed out. Other calculations must be performed manually.

Spherical or Extruded Particles

Calculate the average crush strength of the sample, N (see Note 2), on an as-received basis, reported to the nearest whole number, using the following formula:

$$C = \frac{S}{30} \quad (1)$$

where:

C = average crush strength, N

S = sum of the recorded strengths for the particles crushed, N

30 = number of particles crushed

Calculate the percent particles having a crush strength below 22 N (5 lbf) (see Note 3) reported to the nearest percent, as follows:

$$B = \frac{100P}{30} \quad (2)$$

where:

B = particles having a crush strength below 22 N, %

P = number of particles crushed having a crush strength below 22 N

30 = number of particles crushed

100 = factor to convert to percent

The crush strength data from the 30 particles may be graphically displayed as a histogram. The data are manually transferred to a personal computer (PC) equipped with appropriate software to perform the calculation. A typical histogram is shown in Fig. 3.

Extruded Particles

Calculate the average crush strength per millimeter for extruded particles (see Note 1), reported to the nearest whole number, using the following formula:

$$M = \frac{C}{2} \quad (3)$$

where:

C = previously defined, Eq. 1

M = average crush strength per millimeter for extruded particles, N/mm

2 = contact width of circular anvil, mm

REPORT

Spherical or Extruded Particles

Average crush strength, as calculated in Eq. 1.

Percent particles having a crush strength lower than 22 N, as calculated in Eq. 2.

Maximum crush strength—choose the highest value from the 30 particles crushed and report to the nearest whole number.

Minimum crush strength—choose the lowest value from the 30 particles crushed and report to the nearest whole number.

Extruded Particles

Average crush strength per millimeter, as calculated in Eq. 3, or crush strength per piece (see Note 1).

NOTES

1. The use of semicircular anvils is required for extrudates to minimize length effects by providing a small point of contact with the particle. This results in a true crush rather than a fracture that would typically occur with a flat anvil and an irregularly shaped extrudate. Because the contact width of the semicircular anvil has been determined to be approximately 2 mm, a crush strength per millimeter may be calculated. The average crush strength for whole pieces can be determined, if the length (mm) of each piece is measured before crush, by multiplying the average crush strength per millimeter by the average length in millimeters.
2. Newtons can be converted to lbs_f by multiplying the value obtained in Newtons by 0.2248.
3. The percent particles having a crush strength below 44 N (10 lb_f) can also be calculated.

PRECISION

Repeatability

Based on two tests performed by each of two analysts, on each of two days (8 tests) in one laboratory, the within-laboratory estimated standard deviations (esd) were calculated for crush strength of spherical and extruded catalysts and are listed in Tables 1 and 2. Two tests performed in one laboratory by different analysts on different days should not differ by more than the allowable difference values listed in the Tables at the levels noted (95% probability).

Table 1

Extruded Catalyst

Average Crushing Strength, N/mm	Within-Lab esd, N/mm	Allowable Difference, N/mm
11	1.6	5
9	0.9	3
29	5.0	17

Table 2

Spherical Catalyst

Average Crushing Strength, N	Within-Lab esd, N	Allowable Difference, N
32	3.6	12
54	3.2	11
85	4.9	16

Reproducibility

There is insufficient data to calculate the reproducibility of the test at this time.

TIME FOR ANALYSIS

The elapsed time and labor requirement are identical, 0.15 hour.

REFERENCE

Erweka Instrument Instruction Manual

SUGGESTED SUPPLIERS

Boise-Cascade, 800 W. Bryn Mawr Ave., Itasca, IL 60143 (708-773-5100)
 Erweka Instrument Corp., 56 Quirk Road, Milford, CT 06460 (203-877-8477)
 Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219 (412-562-8300)
 McMaster Carr, P.O. Box 4355, Chicago, IL 60680-4355 (708-833-0300)
 UOP, Monirex Systems, 25 E. Algonquin Rd., Des Plaines, IL 60017-5017 (708-391-2529)

Test #	Newtons
30	54
29	40
28	41
27	47
26	52
25	70
24	47
23	69
22	44
21	70
20	71
19	30
18	38
17	73
16	64
15	45
14	39
13	48
12	81
11	58
10	40
9	63
8	49
7	79
6	49
5	66
4	46
3	68
2	51
1	59

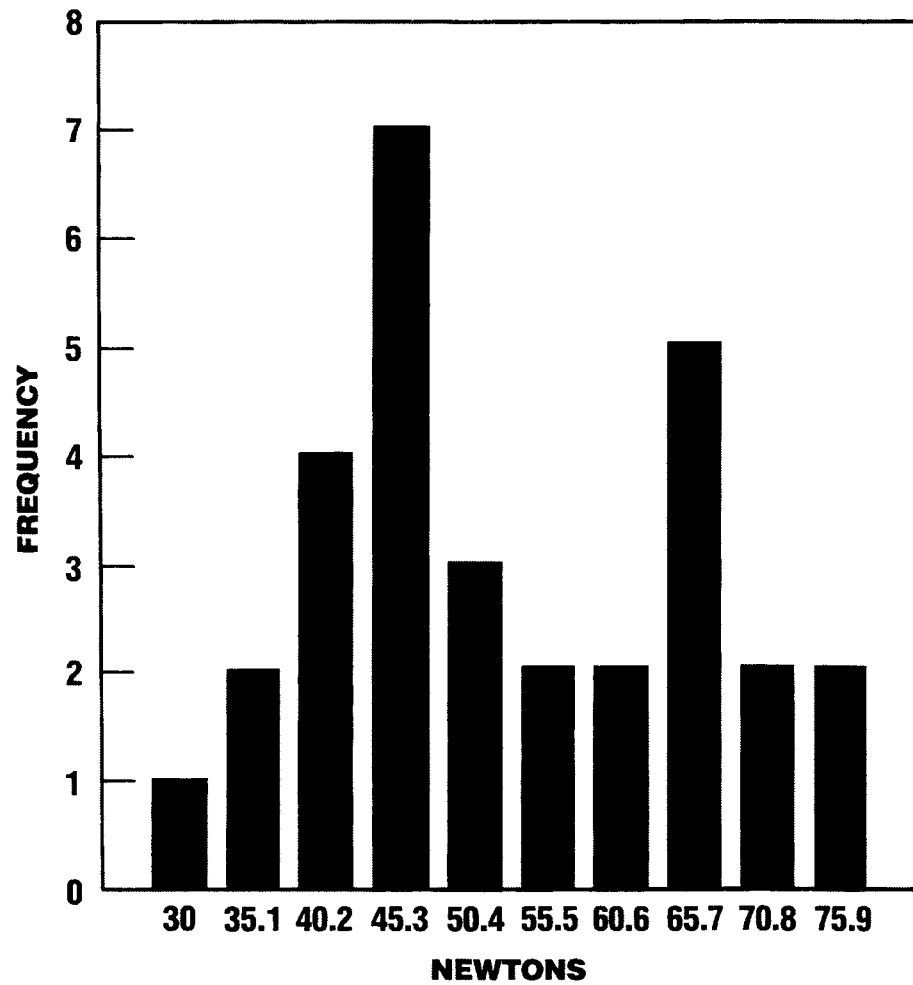


Figure 3
Typical Histogram