

AUTOMATED CRUSH STRENGTH OF EXTRUDED CATALYSTS

UOP Method 973-01

SCOPE

This method is for determining the crush strength of extruded and shaped catalysts using a Chatillon motorized sample stand and force gauge with a chisel anvil. The measurement range is from 1 Newton (N) (0.22 lb_f) to 250 N (56 lb_f). The range can be extended to 450 N (101 lb_f) when using an alternative force gauge. Test apparatus other than the Chatillon may be used provided comparable data can be obtained. For statistical purposes, the number of extruded pieces crushed is set at a minimum of fifty; however, more may be specified.

Data generated using this method cannot be directly compared to those generated using UOP Method 914, Automated Crush Strength of Catalysts or Molecular Sieves. This method will yield higher values for crush strength. Proper comparisons can only be made after tests are run to determine the offset between the two methods. Such offsets are expected to vary from product to product.

OUTLINE OF METHOD

A representative sample is riffled to obtain approximately, but not less than, fifty extrudates. This riffled sample is treated at 500°C (930°F) in a dish for one hour and then transferred in the dish to a hot plate set at 200°C (400°F) to avoid moisture pick-up. Each extrudate is placed on a flat plate at a perpendicular orientation to the chisel attachment that is 1 mm across at the contact point. It is then subjected to a compressive load, and the force required to crush the extrudate is recorded. The procedure is repeated until a minimum of fifty extrudates in the riffled sample have been crushed, and the average crush strength per extrudate is calculated. The percent of extrudates having a crush strength above or below a given value (product specification or customer driven) can be reported, as well as the maximum and minimum crush strength for individual extrudates within the set. In addition, the crush strength data can be graphically displayed as a histogram. Results can be reported in either Newtons (N) or pounds-force (lb_f) as measured using a 1-mm chisel.

APPARATUS

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

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

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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Calibration weights, Class 3, Fisher Scientific, Cat. No. 02-225-26P

Computer, personal, for running Chatillon software, and capable of running Excel™, Minitab™ or similar software to develop a histogram and perform post-analysis calculations, JLW Instrument

Dish, porcelain, Coors 60230, Fisher Scientific, Cat. No. 08-693A

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

Hot plate, capable of 200°C

Muffle furnace, capable of 500°C

Printer, any model compatible with the computer

Riffler, Fisher Scientific, Cat. No. 04-942D

Sieves, wire cloth, brass frame, 8-inch diameter, 2-inch depth, U.S. Standard sieves Nos. 8, 10, 12, 16, 20, and 30, Fisher Scientific, Cat. Nos. 04-881F, G, H, K, M, and P, respectively; with cover and pan, Fisher Scientific, Cat. Nos. 04-886A and B, respectively. The sieves meet current ASTM E 11 and ISO 565/3310-1 specifications.

Software (optional), Minitab or other equivalent for displaying histogram, and Excel or equivalent for post-analysis calculations

Software (required), Chatillon, AutoTest™, to control TCD-200 and collect data from DFA50/100

Surface temperature probe, Spot Check Thermometer, PTC Instruments, Model 573F

Test stand and gauge, Chatillon, includes Cat. No. TCD-200 Motorized Test Stand, Cat. No. DFA50 Turbo gauge including 1 x 6-mm chisel anvil tip, Cat. No. DFA100 Turbo gauge (optional), JLW Instrument

Tongs, crucible, Fisher Scientific, Cat. No. 15-207

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

REAGENTS AND MATERIALS

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

Reference catalysts, extrudate, the same types as samples routinely run, UOP

PROCEDURE

The Chatillon instrument manual must be thoroughly understood before operation or calibration. The instrument must be validated per Chatillon instructions.

Read and understand DFA50, or DFA100, force gauge limits, and TCD-200 operational settings, overloads, etc.

It is recommended that the user select one or more extrudate reference catalysts, similar to those being analyzed, for weekly quality control evaluation (in weeks when samples are being analyzed). Sufficient reference material should be obtained to last for at least six months, preferably one to five years. 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.

Validate instrument performance per the manufacturer's instructions monthly when samples are being analyzed, or when indicated by rule violations, trend shifts, or out of control SPC data. Instrument validation is accomplished by inverting the force gauge and placing known weights on the platform and reading the applied force on the gauge LED. The gauge should be sent to the manufacturer or approved representative yearly for calibration and certification.

- Weight calibration tolerances: 0.5 kg: 4.9 ± 0.4 N, 1 kg: 9.8 ± 0.3 N, 2 kg: 19.6 ± 0.3 N.

Instrument Setup

1. Set the Chatillon reference point to 18.5 mm, following manufacturer's instructions. The reference point and travel distance may vary from site to site. Statistical equivalence must be determined before applying parameter variations.
2. Install the chisel anvil.
3. Select the Chisel Alignment program to mark the flush contact point of the plate and chisel tip, see Table 1. Use a pencil to mark a heavy line on the base plate along the front and back long edge of the chisel. If desired, after the force gauge is raised, mark a line perpendicular to the reference mark just made. This will help align the extrudate catalyst perpendicular to the chisel point.
4. Run zero position program (Z-RECAL, see Table 1) that is appropriate to the method being used.
5. Select and run appropriate analysis program. Table 1 lists general parameter settings for the Chatillon instrument. Other instrument parameters may be used, based upon the sample type. It is the laboratory's responsibility to show correlation with the general parameter settings.
 - To avoid confusion, the parameters in Table 1 are listed as they appear on the instrument screen. Not all parameters are used.
 - *Test code* and *Test name* designations may differ from site to site.

Sample Analysis

1. Remove fines and small pieces by placing the sample onto one of the sieves listed in Table 2, depending upon the nominal diameter of the product. Shake gently for one minute.
2. Riffle the screened extrudates down to a sub-sample size of approximately, but not less than, 50 extrudates.
3. Transfer the riffled extrudates to a dish and place in a 500°C muffle furnace for one hour.
 - *CAUTION: Wear appropriate personal protective equipment and use tongs when transferring dish to the muffle furnace and from the muffle furnace to the hot plate.*
 - Tests have shown that crush strength decreases with increasing sample moisture content.

Table 1
General Chatillon Parameter Settings

	<u>Standard Extrudate</u>	<u>Zero Recalibration</u>	<u>Chisel Alignment</u>
Test type #	1	2	3
Test Code	UOP-120	Z-RECAL	Chisel
Test Name	Crush Test	Recalibrate Zero Pt	
Sample quantity	55	0	0
Xname	Deflection	X	X
Xunits	mm	mm	mm
Speeds	120	3	3
Home deflection	0	18.3	0.5
Begin deflection	-12	0	0
End deflection	-17.5	0	0
Yname	Force	Y	Y
Yunits	N	N	N
Min force	1.5	0	0
Max force	200	0	0
Pollprog	Addpol0	Zz-recal	Zz-recal
Failure	-0.7	0	0
Aux value	n/a	0	0
Min x interval	-1	0	0
Cycle profile	n/a	n/a	n/a
Control profile	n/a	n/a	n/a

Table 2
Sieves for Screening of Sample

<u>Nominal Diameter (mm)</u>	<u>U.S. Standard Sieve No.</u>
Below 1.3	30
1.3	20
1.6	16
2.1	12
2.5	10
Above 2.5	8

4. Quickly transfer the dish to a hot plate set at a minimum of 200°C.
 - Begin the crush strength determinations without delay, and continue until the required number of extrudates are crushed.
5. Run the zero recalibration (Z-RECAL) program between each sample to prevent any program problems.
6. Position a hot extrudate at the center of the perpendicular lines using the blunt forceps.
 - *CAUTION: Take precautions to avoid burns when picking up an extrudate from the dish on the hot plate.*
7. Press "ENTER" to begin movement of the motor-driven gauge.

- After the extrudate is crushed, the moveable anvil automatically returns to the preset position.
8. Brush the crushed material from the anvils into the utility tray with the camel's hair brush.
 9. Insert the next extrudate immediately.
 10. Repeat Steps 6 through 9 until a minimum of 50 extrudates riffled from the original sample have been crushed (testing of a larger number of extrudates may be requested).

CALCULATIONS

The calculation of average crush strength is pre-programmed into the AutoTest software. Report values to the nearest whole number if in N, or the nearest tenth if in lb_f. Multiply the raw N value from the instrument printout times 0.2248 to convert to lb_f. Eq. 1 is the manual calculation of average crush strength. Reports can be configured and saved for later use.

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

where:

C = average crush strength, N or lb_f

S = sum of the recorded strengths for all extrudates crushed, N or lb_f

M = width in mm of the chisel piece used, 1 mm for the stock chisel piece

X = number of extrudates crushed

If requested, calculate the percent of extrudates having crush strength above or below a given value and report to the nearest tenth of a percent using Eq. 2. See the specification for the particular product or customer.

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

where:

B = extrudates having a crush strength above or below a given value, %

P = number of extrudates crushed having a crush strength above or below a given value

X = previously defined, Eq. 1

100 = factor to convert to percent

If further evaluation is required, transfer the data to the personal computer equipped with Excel or Minitab to perform the calculations and display a histogram.

REPORT

Report all values listed below in N to the nearest whole number, or in lb_f to the nearest tenth.

- Average crush strength, as calculated in Eq.1 (or from the instrument printout).
- Percent of extrudates having a crush strength above or below a given value, calculated in Eq. 2.
- Maximum crush strength - choose the highest value from the extrudates crushed.
- Minimum crush strength - choose the lowest value from the extrudates crushed.

PRECISION

The precision statements were determined using UOP Method 999.

A nested design was carried out for determining crush strength of three reference extruded catalysts with two analysts in three laboratories. Each analyst carried out tests on two separate days, performing two tests each day for each sample. The total number of tests performed was 72. The precision data are summarized in Table 3. Two tests performed by the same analyst on the same day should not differ by more than the ASTM allowable difference shown in Table 3 with 95% confidence. Two tests performed in one laboratory by different analysts on different days should not differ by more than the UOP allowable difference shown in Table 3 with 95% confidence. Two tests performed by different analysts in different laboratories on different days should not differ by more than the Reproducibility allowable difference shown in Table 3 with 95% confidence.

Table 3
ASTM and UOP Repeatability and Reproducibility, Newtons

Sample	Mean	<u>ASTM Repeatability</u>		<u>UOP Repeatability</u>		<u>Reproducibility</u>	
		Within-Day esd	Allowable Difference	Within-Lab esd	Allowable Difference	Within-Lab and Between-Lab esd	Allowable Difference
1	26	1.0	3	1.5	5	1.8	6
2	41	1.9	6	2.3	7	2.9	10
3 (Trilobe)	28	1.9	6	1.8	5	3.5	14

The data in Table 3 are a short-term estimate of repeatability. When the test is run routinely, a control standard and chart should be used to develop a better estimate of the long-term repeatability.

TIME FOR ANALYSIS

The elapsed time for a single analysis is 1.8 hours, with a labor requirement of 0.8 hour.

REFERENCES

Ametek DFA50 Force Gauge Manual, included with instrument
 AutoTest Software Manual, included with instrument
 Chatillon Instrument Manual, included with instrument
 UOP Method 914, "Automated Crush Strength of Catalysts or Molecular Sieves"
 UOP Method 999, "Precision Statements in UOP Methods"

SUGGESTED SUPPLIERS

JLW Instrument Corp., 452 N. Sangamon, Chicago, IL 60622 (312-666-0595) jlwinstruments.com
 Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219 (412-562-8300) fishersci.com
 PTC Instruments, 2301 Federal Ave., Los Angeles, CA 90064-1482 (310-312-0826)