



A Honeywell Company

Length and Diameter of Extruded Catalyst Using a Semi-Automated Method

UOP Method 947-15

Scope

This method is for determining the mean length (L) and diameter (D) of fresh extruded catalyst and extruded catalyst supports. Catalysts of various diameters and lengths can be measured. The mean length and diameter can be reported in either inches or millimeters. The data can also be reported as the ratio of length over diameter, L/D. Fines, if present, are disregarded.

References

Ono Sokki Handbook

UOP Method 999, "Precision Statements in UOP Methods," www.astm.org

Outline of Method

The length and diameter of thirty individual pieces of catalyst are measured, using the Ono Sokki Linear Gauge, a semi-automated device. A built-in microprocessor calculates the means of the thirty length and diameter measurements which are reported in either inches or millimeters. A simple calculation yields the length over diameter ratio, if required. Larger sets of catalyst can be used (i.e., fifty or one hundred pieces, etc.), but this will lengthen the time for analysis.

Apparatus

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

Blocks, calibration, 0.0625- and 0.250-inch, micrometer calibration set, Fowler, Cat. No. 53-670-002

Forceps, dressing, 114-mm length, Fisher Scientific, Cat. No. 13-812-39

Gauge, linear, Ono Sokki, Model EG-225

Printer, Ono Sokki, Model RQ-2110

Stand, gauge, Ono Sokki, Model ST-022

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. SAFETY DATA SHEETS (SDS) OR EXPERIMENTAL SAFETY DATA SHEETS (ESDS) 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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Reagents and Materials

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

Finger cots, Fisher Scientific, Cat. No. 11-392-8B

Forceps, plastic, disposable, Fisher Scientific, Cat. No. 10-309

Methanol, Fisher Scientific, Cat. No. A-454-1

Tissues, labwipes, Fisher Scientific, Cat. No. 06-666-1A

Calibration

In order to verify that the Ono Sokki gauge is giving accurate data, it must be checked with known calibration gauge blocks. This calibration should be checked a minimum of once per week with the two available gauge blocks, during periods when samples are being analyzed. It is important that the gauge blocks are clean and free of fingerprints.

1. Clean the calibration blocks with an alcohol-damp labwipe prior to calibration to ensure that the blocks are clean and free of fingerprints. The gauge blocks should be handled with plastic forceps or latex finger cots. If finger cots are used, they should be cleaned with an methanol-damp labwipe after they are placed over the fingers.
2. Clean the granite base and the spindle on the Ono Sokki with an methanol-damp labwipe.
3. Turn on the Ono Sokki EG-225 gauge and the RQ-2110 printer.
4. Put the unit measurement switch in the “inch” position.
 - The instrument can alternatively be set to measure in millimeters (mm).
5. Press the EG-225 red reset button with the spindle contact tip resting on the base platform. Make sure the contact tip is touching the platform top. The display should read all zeroes.
 - The instrument is now zeroed and ready to take readings. It is not necessary to reset the instrument during the test run. If you are in doubt about whether the instrument is zeroed, repeat Step 5 above and continue the test.
6. Lift the spindle with the finger lift to clear the gauge block to be measured.
7. Position the gauge block to be measured under the spindle.
8. Bring the spindle to the top of the gauge block and gently release the finger lift.
9. Take the reading from the gauge and recorder.
10. Repeat Steps 1 through 7 for the second block and any subsequent checks.
 - The gauge blocks should be stored wrapped in labwipes or Kleenex. Treat the blocks with utmost care.
 - Prolonged contact with body heat or other heat sources can expand the block and compromise the calibration.
 - If the calibration check is ± 0.0004 inch outside of 0.0625 inch, or ± 0.0007 inch outside of 0.250 inch do not run samples, attempt to locate and correct the problem.

Procedure

The analyst is expected to be familiar with general laboratory practices, the technique of gauge measurement, and the equipment being used. Dispose of used supplies and samples in an environmentally safe manner according to applicable regulations.

Obtain thirty random pieces of catalyst from the sample to be measured. Measure all thirty pieces for diameter and then a second time for length; the length and diameter measurements cannot be interspersed.

1. Turn on the Ono Sokki EG-225 gauge and the RQ-2110 printer.
2. Put the unit measurement switch in the "inch" position.
 - The instrument can alternatively be set to measure in millimeters (mm).
3. Press the EG-225 red reset button with the spindle contact tip resting on the base platform. Make sure the contact tip is touching the platform top. The display should read all zeroes.
 - The instrument is now zeroed and ready to take sample readings. It is not necessary to reset the instrument during the test run. If you are in doubt about whether the instrument is zeroed, repeat Step 3 above and continue the test.
4. Lift the spindle and insert one catalyst piece beneath the contact tip for either length or diameter measurement.
5. Bring the spindle contact tip down gently to just contact the catalyst. The reading on the instrument display is the diameter or length in inches (or millimeters if that was chosen in Step 2).
6. Press the printer print button to record the results.
 - An entry may be canceled by pressing the RQ-2110 printer cancel button. The entry will be printed but not included in the final results.
7. Repeat Steps 5 through 7 for the other 29 pieces of catalyst.
8. Press the edit button for the statistical report.
 - The statistical report gives the following:
 - N = number of measurements
 - Max = maximum measurement in set
 - Min = minimum measurement in set
 - R = range (Max minus Min)
 - X = average
 - σ = estimated standard deviation (esd)
9. Press the printer reset button to clear the instrument for the other dimensional measurement, or for the next catalyst set.
 - The contact tip and platform must be kept clean. Do not handle with the fingers. Always lift and lower the spindle with the finger lifter. If the spindle becomes dirty and does not function smoothly, wipe it with a cloth wetted with methanol. Other organic solvents may remove the paint.

Calculations

The instrument calculates and prints out the mean values for length (L) and diameter (D). Report them to no more than three significant figures (limited to no more than four places after the decimal). L and D are reported in either inches or millimeters (mm). Calculate the ratio of L over D by dividing the mean length by the mean diameter. Report L/D to the same number of significant figures as the lesser number of significant figures in the L or D measurement (typically three) used in the calculation. Report the result as L/D ratio (no units).

Precision

Precision statements were determined using UOP Method 999, "Precision Statements in UOP Methods."

Repeatability and Site Precision

A nested design was carried out for determining L/D on three catalyst types by two analysts on two separate days, performing two analyses each day for a total of 24 analyses. Using a stepwise analysis of variance procedure, the within-day estimated standard deviations (esd) were calculated at the concentration means listed in Table 1. Two analyses performed in one laboratory by the same analyst on the same day should not differ by more than the repeatability allowable differences shown in Table 2 with 95% confidence. Two analyses performed in one laboratory by different analysts on different days should not differ by more than the site precision allowable differences shown in Table 2 with 95% confidence.

Table 1
Repeatability and Site Precision, inches

<u>Parameter</u>	<u>Mean</u>	<u>Repeatability</u>		<u>Site Precision</u>	
		<u>esd</u>	<u>Allowable Difference</u>	<u>esd</u>	<u>Allowable Difference</u>
Sample A					
Length, inches	0.213	0.0097	0.038	0.0165	0.100
Diameter, inches	0.058	0.0005	0.002	0.0006	0.003
L/D Ratio	3.696	0.1663	0.653	0.2892	1.760
Sample B					
Length, inches	0.196	0.0131	0.052	0.0155	0.070
Diameter, inches	0.061	0.0004	0.002	0.0004	0.002
L/D Ratio	3.233	0.2363	0.928	0.2741	1.233
Sample C					
Length, inches	0.214	0.0066	0.026	0.0091	0.036
Diameter, inches	0.097	0.0003	0.001	0.0003	0.001
L/D Ratio	2.213	0.0706	0.277	0.0989	0.445

The data in Table 2 represent short-term estimates of the repeatability of the method. When the test is run routinely, use of a control standard and a control chart is recommended to generate an estimate of long-term repeatability.

Reproducibility

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

Time for Analysis

The elapsed time and labor requirement for one sample length and diameter determination are the same, 0.5 hour.

Suggested Suppliers

Fisher Scientific, 300 Industry Dr., Pittsburgh, PA 15275, USA, 1-412-490-8300,
www.fishersci.com

Fowler High Precision, 66 Rowe St., Newton, MA 02466, USA, 1-617-332-7004,
www.fvfowler.com

Ono Sokki Technology Inc., 2171 Executive Drive Suite 400, Addison, Ill 60101, USA, 1-630-627-9700, www.onosokki.net