



A Honeywell Company

Fines in Catalysts and Catalyst Supports by Sieve Analysis

UOP Method 333-10

Scope

This method is for determining the fines content of catalysts or catalyst supports (spheres, extrudates, and trilobes) by screening through appropriate USA Standard Series testing sieves.

References

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

Outline of Method

A representative sample is placed on top of a nest of appropriate mesh size sieves with a bottom pan. The nested sieves are placed in a sieve shaker, covered, and shaken with a specific throwing motion with angular momentum for ten minutes. The material retained on each sieve and in the bottom pan receiver is collected and weighed individually. The mass percent of material in the bottom pan, and, optionally, on the last sieve, is then calculated and reported as percent fines.

Apparatus

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

Balance, readability 0.01 gram, with digital interface. See the shaker manufacturer's software or representative for balance compatibility.

Bottom pan, sieve, stainless steel, 20.3-cm (8-inch) diameter, 5.1-cm (2-inch) height, Retsch, VWR, Cat. No. 66004-848

Brush, paint, VWR, Cat. No. 17230-060

Computer, compatible with shaker interface and software

Riffler, sample splitter, VWR, Cat. No. 56723-250

Sieve shaker, with clamp cover, Retsch AS 200 Control with Easy Clamp, VWR Cat. No. 66004-956. Easy Sieve software is included. This sieve shaker employs a specific throwing motion with angular momentum. Different results may be obtained if other sieve shakers are used. Substitution is permitted only if equivalent results are demonstrated.

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Sieves, USA Standard designation, stainless steel wire cloth, stainless steel frame, 20.3-cm (8-inch) diameter, 5.1-cm (2-inch) height. Sieve sizes and nest designations are determined by the size of the material to be tested and any specifications related to the material. Table 1 is a list of suggested sieves to be used. Sieves may be added or deleted depending upon the materials to be tested and test requirements. Important: If the results will be compared between laboratories, the sieves must be “Matched” test sieves from the same manufacturer.

Table 1
Part Numbers for Sieves

<u>USA Standard</u>	<u>Size Opening</u>	<u>VWR Part Number</u>
No. 8	2.36 mm	14212-122
No. 10	2.00 mm	66004-896
No. 12	1.70 mm	66004-894
No. 14	1.40 mm	66004-892
No. 16	1.18 mm	66004-890
No. 18	1.00 mm	66004-888
No. 20	0.85 mm	66004-886

Procedure

The analyst is expected to be familiar with general laboratory practices and the equipment being used.

While the procedure specifies the use of a No. 16 sieve, different types of catalyst or catalyst supports may require different sieve sizes. Please refer to the catalyst specifications prior to running this test. In addition, some catalyst specifications may require two fines measurements, one being the fines in the pan, and the other on the last sieve. The steps below are written specifically for the Retsch AS 200 Control model of shaker listed in *Apparatus*.

- Using approximately 500 grams of sample, riffle to obtain a 50 to 75 gram representative sample of the material to be tested.
 - Follow instructions supplied with the riffler for proper riffing procedure.
- Open the Easy Sieve program. Click on Sieve Analysis in the menu bar. Choose one of the programs under the task file list from the drop down menu. Edit the parameters and sample information and press “OK.” The parameters listed in Table 2 have been found suitable for a range of samples.
 - Please refer to the manual for the Retsch Sieve Shaker prior to starting an analysis for operating instructions and for editing the screens from the program.

Table 2
Sieve Shaker Parameters

Bottom sieve screen size	No. 16
Amplitude	1
Time	10 min
Sample size	50 g

- Tare the balance and place the empty bottom pan on the balance. Press the “PRINT” button on the balance to transmit the weight of the pan to the program.
- Tare the balance and place the finest mesh sieve on the bottom pan. Press the “PRINT” button on the balance to transmit the weight of the screen to the program. Repeat for each additional screen

used for the analysis.

5. Tare the balance and place the sample onto the top screen. Press “PRINT” to transmit the weight of the sample. Remove screens from the balance and place on the Retsch sieve shaker. Attach and secure the screens with the clamp cover. Press “OK” on the screen prompts to start sieving.
6. Re-zero the balance.
7. When the analysis is finished, loosen the clamps on the cover, remove the top screen, and place on the balance. Press “PRINT” to transmit weight to appropriate place in the program. Repeat for each screen until all the screens are complete. When finished, click on the “CALCULATE” button on the screen. For a table of numeric data, go to “RESULTS → TABLE” or simply press “F5.”

Calculations

Calculations are performed by the data system

Report

Report results to one decimal place as follows:

Fines, result, mass-%; then state the sieve used, e.g., through USA No. 16

Precision

Precision statements were determined using UOP Method 999.

Repeatability and Site Precision

A nested design was carried out for determining the fines in six catalyst support samples with two analysts in one laboratory. Each analyst carried out tests on two separate days, performing two tests on each sample each day. In addition, Alumina Support #4 was analyzed in a second laboratory using the same protocols. Data from the second laboratory are included in the precision calculations. The total number of tests was 56. 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 repeatability allowable difference with 95% confidence. Two tests performed in one laboratory by different analysts on different days should not differ by more than the site precision allowable difference with 95% confidence.

Table 3
Repeatability and Site Precision, Fines, mass-%

<u>Sample</u>	<u>Mean</u>	<u>Repeatability</u>		<u>Site Precision</u>	
		<u>Within- Day esd</u>	<u>Allowable Difference</u>	<u>Within- Lab esd</u>	<u>Allowable Difference</u>
Alumina Support #1	0.2	0.05	0.2	0.06	0.2
Alumina Support #2	0.5	0.04	0.1	0.04	0.1
Alumina Support #3	0.6	0.04	0.1	0.05	0.2
Alumina Support #4	1.7	0.07	0.2	0.10	0.3
Charcoal Support #1	1.0	0.23	0.9	0.28	1.1
Charcoal Support #2	1.7	0.23	0.9	0.29	1.1

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.

Reproducibility

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

Time for Analysis

The elapsed time is 0.9 hour; with a labor requirement of 0.5 hour.

Suggested Suppliers

VWR International is listed as the supplier for all of the apparatus including the Retsch sieve shaker and related parts. Alternatively, the sieve shaker and related parts may be purchased directly from Retsch.

Retsch Inc., 74 Walker Lane, Newton, PA 18940 USA (267-757-0351) www.retsch.com

VWR International, 1310 Goshen Parkway, West Chester, PA 19380 USA (610-431-1700)
www.vwr.com