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Standard Test Method for
Particle Size Distribution of Catalytic Materials by Sieving[1]

This standard is issued under the fixed designation D4513; 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 particle size distribution of catalytic powder material using a sieving instrument and is one of several found valuable for the measurement of particle size. This test method is particularly suitable for particles in the 20 to 420-µm range.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

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]

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
E161 Specification for Precision Electroformed Sieves
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E456 Terminology Relating to Quality and Statistics

3. Summary of Test Method

3.1 A 50 % relative humidity-equilibrated sample of known weight is allowed to fractionate on a series of various size sieves to allow the various particle sizes to be collected on successively smaller sieve openings.

3.2 The sample fraction collected on each sieve of the series is weighed and its fractional part of the original sample is determined.

4. Significance and Use

4.1 This test method can be used to determine particle size distributions of catalysts and supports for materials specifications, manufacturing control, and research and development work.

5. Apparatus

5.1 *Laboratory Sieving Instrument*, automatic with timer preferred.

5.2 *U.S. Standard Sieves*, or equivalent, to include micrometres (mesh) 420(40), 250(60), 177(80), 149(100), 105(140), 74(200), 44(325) and electroformed 30 and 20 micrometres. Because of their superior uniformity and resistance to distortion or damage during use, electroformed sieves, preferably with square holes, are recommended. Sieves with diameters between 6 and 10 cm are suggested.

5.3 *Ultrasonic Cleaning Tank*, 100 W.

5.4 *Transmitted Light Microscope*, 300 magnification, with calibrated scale eyepiece.

5.5 *Heat Gun Dryer*, (hair dryer or equivalent).

5.6 *Analytical Balance*, capable of weighing to 0.001 g.

5.7 *Sample Splitter, Chute Type, or Spinning Riffler*, with spinning riffler preferred.

6. Reagents

6.1 *Antistatic Coating*, (record cleaning spray or equivalent.)

6.2 *Alcohol-Water Solution*—One part ethanol to nine parts deionized or distilled water.

7. Sampling

7.1 The sample must be free-flowing and homogeneous. If particle size segregation is apparent to either the eye or from observation under a microscope, remix and resample the material using the proper riffling procedure.

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[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.

7.2 Equilibrate the sample at 20 to 25°C (68 to 77°F) in a desiccator with a humidity level of 50 %. A 24-h period is usually sufficient.

8. Calibration and Standardization

8.1 Prior to use, check all sieves for damage or improper cleaning. If woven-wire sieves are used rather than the preferred electroformed sieves, it is especially important to carefully inspect the wire surface for wear, misalignment, tears, creases, or separation along the edges.

NOTE 1—Specifications for wire cloth sieves are described in Specification E11 and specifications for electroformed sieves are described in Specification E161.

9. Procedure

9.1 Select appropriate sieves for the sample being analyzed, typically the 149, 105, 74, 44, and 20-µm sieves.

NOTE 2—For optimum results, the estimated particle size should be determined by microscopic examination at 100–300X. Sieves may then be selected to cover the size range of the particles.

9.2 Clean 44 and 20-µm sieves prior to use in an ultrasonic bath using a 10 % ethanol, 90 % water mixture. Dry the sieves in a low temperature air jet (hair dryer or equivalent) and allow to equilibrate at room temperature for 30 min before obtaining the tare weights.

9.3 Tare each sieve and the fines collector pan, recording each weight to the nearest 0.001 g.

9.4 After taring, moisten a sheet of tissue paper with antistatic spray and coat the inside wall surface of each sieve by rubbing with the coated tissue.

9.5 Place the sieves in a vertical stack in descending order by mesh size (largest on top).

9.6 Weigh a suitable amount of sample obtained by riffling, normally 0.5 to 1.0 g, and transfer into the largest mesh sieve at the top of the stack.

9.7 Complete the assembly of the apparatus.

9.8 Turn on and adjust to provide rapid transport through the sieves.

9.9 Continue sieving for 2 min after no further separation is detectable.

NOTE 3—After completion of sieving, none of the sieves should contain more than two to three particle layers. For most powder samples, 0.5 g of sample provides a satisfactory quantity distribution.

9.10 Stop the sieve action.

9.11 Remove sieves carefully and weigh each sieve and the pan separately. Note the gross weight for each one and record above the corresponding tare weight.

9.12 Sum the weight of sample on each sieve and the pan to obtain the total weight of the recovered sample. The total weight of recovered material should check within 5 mg of the starting sample weight.

NOTE 4—Examine the sieve fractions under a microscope to determine whether the sieve particles in each fraction are within the size range between the sieve and the next coarser sieve. If appreciable finer or coarser particles are present, tackiness is indicated. Dry and reequilibrate the sample and repeat the analysis.

TABLE 1 Presentation of Data Weight of Sample Used, 0.610 g

Sieve No.	Mesh Size microns	Net Weight, g	Weight % Sieve Fraction	Cumulative % Passing
100	149	0.037	6.1	93.9
140	105	0.034	5.6	88.3
200	74	0.083	13.6	74.7
325	44	0.193	31.6	43.1
635	20	0.196	32.1	11.0
Pan		0.067	11.0	
Total weight recovered		0.610	100.0	

10. Presentation

10.1 Calculate the weight percent of sample on each sieve by multiplying the net weight of each fraction by 100 and dividing by the total weight the total weight of recovered sample.

$$\text{Weight \% sieve fraction} = 100 \times (S - T)/W$$

where:

S = total weight after sieving, g,
T = tare weight of sieve, g, and
W = total weight of recovered sample, g.

10.1.1 Calculate the cumulative percentage passing through each sieve by adding its fractional percentage to the fractional percentage of all coarser sieves, and subtracting the total from 100 %. See Table 1 for an example of the calculations and presentation.

10.2 *Median Particle Size*—The median particle size may be determined by plotting the cumulative percentage data against the mesh size and determining the size corresponding to 50 %.

11. Precision and Bias (Note 5)

11.1 Agreement among individual measurements was determined using an equilibrium fluid cracking catalyst. Experimental repeatability was measured for a number of analyses in each of five laboratories. Experimental reproducibility was determined by comparison of results from all seven of the laboratories participating in the round-robin testing program. Pairs of test results obtained by a procedure similar to that described herein are expected to differ in absolute value by less than 2.77*S*, where 2.77*S* is the 95 % probability limit on the difference between two test results, and *S* is the appropriate estimate of standard deviation.

NOTE 5—Use of the terms "repeatability," "reproducibility," "precision," and "bias" are in accordance with Terminology E456 and Practice E177.

11.1.1 *Experimental Repeatability*[3]—Repeatability is used to designate the ability of an instrument to report the same answer assuming no sample bias or operator influence. A measure of instrument repeatability is the standard deviation of a number of runs. The results of testing the equilibrium fluid catalytic cracking catalyst sample in each of five laboratories produced an average standard deviation of the interpolated

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[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D32-1015.