Designation: ~~D4513 – 11 (Reapproved 2017)~~ D4513 – 22

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~~420 μm range. (See Terminology D3766.)

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.2.1 *Exception*—In 5.2, mesh size is the standard unit of measure.

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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3766 Terminology Relating to Catalysts and Catalysis
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
E105 Guide for Probability Sampling of Materials
E161 Specification for Electroformed Material and Test 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.

[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.

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) 425(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~~ 10 cm are suggested.

5.3 *Ultrasonic Cleaning Tank,* ~~100 W.~~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.~~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.)~~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~~ Obtain a representative sample of material from larger composites by riffling or splitting in accordance with subsection 5.12 of STP 447A,[3] ~~size segregation is apparent to either the eye or from observation under a microscope, remix and~~ or some other suitable means, with the aim of obtaining a sample that represents the size distribution of the larger composite. The analyst should also consult Guide E105 ~~resample the material using the proper riffling procedure.~~to help develop a sampling plan.

7.1.1 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.

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

[3] STP 447A, *Manual on Test Sieving Methods*, ASTM International, West Conshohocken, PA 19428.

9. Procedure

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

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:*~~
where:

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