Designation: D8413 – 21

Standard Guide for Measuring the Water Pore Volume of Catalytic Materials by Centrifuge[1]

This standard is issued under the fixed designation D8413; 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 guide describes how to measure the pore volume of catalytic materials by water immersion with the excess water removed with a centrifuge. The measured pore volume is converted to the dry pore volume by using the loss on ignition (LOI) of the material. It is generally applicable to both powdered materials and particles greater than about 1 mm.

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.

1.3 *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, health, and 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

D4284 Test Method for Determining Pore Volume Distribution of Catalysts and Catalyst Carriers by Mercury Intrusion Porosimetry

D6761 Test Method for Determination of the Total Pore Volume of Catalysts and Catalyst Carriers

E1272 Specification for Laboratory Glass Graduated Cylinders

2.2 *Other ASTM Documents:*[3]

STP 447A Manual on Test Sieving Methods

3. Terminology

3.1 *Definitions*—See Terminology D3766.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *water pore volume, n*—also known as the pore volume determined by water absorption, is a measure of the porosity of a catalytic material.

4. Summary of Guide

4.1 This guide measures pore volume by water immersion with the excess water removed with a centrifuge. The measured pore volume is converted to the dry pore volume by using the LOI of the material.

5. Significance and Use

5.1 This guide provides an alternative way to measure the porosity of catalytic materials without the use of mercury porosimetry. It is useful for research and development as well as quality control purposes. (See Test Methods D4284 and D6761.)

6. Interferences

6.1 There are no known interferences. This method cannot be used for materials that react with or dissolve in water.

7. Apparatus

7.1 *Balance,* accurate to nearest 0.0001 g.

7.2 *Centrifuge Tubes,* custom made according to drawings using the tube described in Fig. 1 for particles >~1 mm or the tube described in Fig. 2 for powders. These tubes can be fabricated from any suitable material, including glasses such as borosilicate glass or plastics such as nylon.

7.3 *Centrifuge,* capable of a force of at least 456 G and equipped with timer, speed control and shields, such as a Dynac 11 or equivalent. (See Note 1.)

7.4 *Beaker,* 1000 ml.

[1] This guide 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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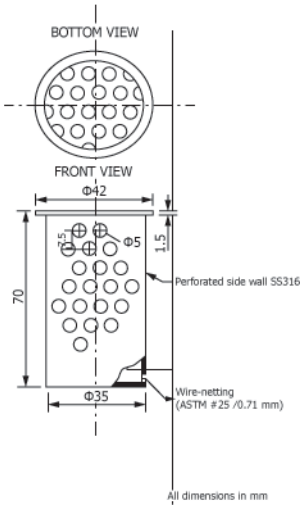
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org.

Figure 1: Water Pore Volume Centrifuge Tube Dimensions



NOTE 1—These tubes can be fabricated from any suitable material, including glasses such as borosilicate glass or plastics such as nylon.

FIG. 1 Sample Holder for >1 mm Particles

7.5 *Graduated Cylinder,* capacity 100 mL, conforming to Specification E1272, Class A.

7.6 *Porcelain Crucible,* 100 ml.

7.7 *Furnace,* capable of 482 °C.

7.8 *Timer.*

8. Reagents and Materials

8.1 Deionized water (DI) or ASTM distilled water type 4 or purer.

9. Hazards

9.1 There are no known hazards to this procedure. The user should use appropriate personal protective equipment and follow good lab practices.

10. Sampling, Test Specimens, and Test Units

10.1 Test samples should be obtained from larger composites by riffling or splitting in accordance with subsection 5.12 of STP 447A or some other suitable means with the aim of obtaining a sample that represents the size distribution of the large composites.

11. Preparation of Apparatus

11.1 Follow the centrifuge maker's recommendations.

12. Calibration and Standardization

12.1 Follow the centrifuge maker's recommendations.

13. Conditioning

13.1 None required except as specified in the procedure.

14. Procedure

14.1 Weigh two dry empty centrifuge tubes to the nearest 0.01 g and record the weight of each (M1). Refer to Figs. 1 and 2 for centrifuge tube requirements.

14.2 Divide 85 ± 10 cc of sample between the two centrifuge tubes. The final weight of the tubes should be within ± 0.2 g of each other.

14.3 Weigh each of the filled tubes to the nearest 0.01 g and record this weight as (M2).

14.4 Set the speed of the centrifuge at the approximate rpm without centrifuge tubes but with all four empty shields in place. (See Note 1.) This speed should be checked daily at the beginning of a set of samples.

NOTE 1—The relative centrifuge force (RCF) should be 456 G. For the Dynac 11, this corresponds to a speed of 1500 rpm. RCF is given by: $Rpm = \sqrt{(RCF / 28.4\ r) \times 1000}$ where r is the top radius in inches of the rotor.

14.5 Slowly place both centrifuge tubes in a beaker of DI water with the sample completely immersed for 15 min. To prevent particles from being lifted out of the tube during immersion, the top layer can be pre-wetted prior to immersion. (See Note 2.)

NOTE 2—Clean DI water should be used for each set of samples.

14.6 Remove the tubes from the beaker and allow the excess water to drain for 1 min.

14.7 Put both tubes in opposite positions in the centrifuge and close the lid.

14.8 Switch the centrifuge on and start the timer.

14.9 Switch off the centrifuge after 1 min (Note 3).

NOTE 3—It will take approximately 20-30 seconds for the centrifuge to reach its preset speed of 1500 rpm. The total time of 60 seconds includes this start up time.

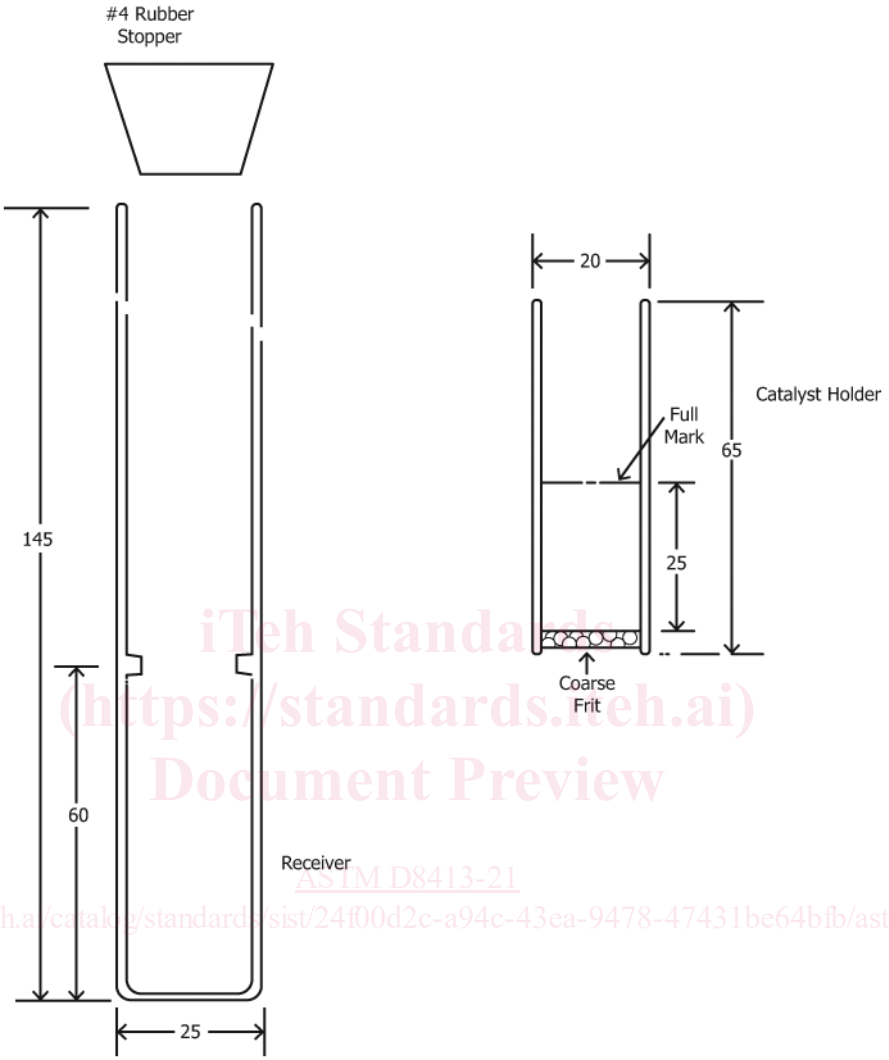
14.10 Reweigh each tube to the nearest 0.01 g (M3).

14.11 Determine the LOI of the "as received" sample.

14.11.1 Dry a porcelain crucible at 482 °C.

14.11.2 Place crucible in desiccator to cool.

14.11.3 Weigh crucible to ± 0.0001 g (WA).



NOTE 1—These tubes can be fabricated from any suitable material, including glasses such as borosilicate glass or plastics such as nylon.

FIG. 2 Sample Holder for Powdered Samples

14.11.4 Add 1-5 g of material and weigh sample and crucible to ± 0.0001 g (WB).

14.11.5 Place crucible in oven and heat to 482 °C and soak for 1 h. If the sample being tested decomposes at 482 °C, a lower temperature can be used and that difference noted in any data reporting.

14.11.6 Remove sample from furnace and cool in desiccator.

14.11.7 Weigh the crucible for sample final weight (WF).

15. Calculation or Interpretation of Results

15.1 *Percent LOI:*

$$\text{LOI} = \frac{\text{WB} - \text{WF}}{\text{WB} - \text{WA}} \times 100 \quad (1)$$

where:

WB = sample and crucible weight,
WA = crucible weight, and
WF = sample and crucible after 482 °C.

15.2 *Pore Volume (As Received):*

$$\text{PV (a r)} = \frac{\text{M3} - \text{M2}}{\text{M2} - \text{M1}} \text{ cc/gm} \quad (2)$$

15.3 *Pore Volume (Dry):*

$$\text{PV (dry)} = \frac{\text{PV (a r)} \times (100 + \%\ \text{LOI})}{100 - \%\ \text{LOI}} \quad (3)$$