Designation: D 6761 – 02

Standard Test Method for Determination of the Total Pore Volume of Catalysts and Catalyst Carriers[1]

This standard is issued under the fixed designation D 6761; 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 the total pore volume of catalysts and catalyst carriers, that is, the volume of pores having pore diameter between approximately 14 µm and 0.4 to 0.6 nm (4 to 6 Å).

1.2 This test method involves hazardous materials, operations and equipment. *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 to determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 8, 9.1.7, and 9.1.11.

2. Referenced Documents

2.1 *ASTM Standards:*

D 3766 Terminology Relating to Catalysts and Catalysis[2]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]

E 456 Terminology Relating to Quality and Statistics[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

3. Terminology

3.1 *Definitions*:

3.1.1 *particle volume*—the volume of a particle including pores into which mercury cannot penetrate at ambient pressure (smaller than approximately 14 µm diameter pore mouth).

3.1.2 *true volume*—the volume of a particle, including pores, into which helium cannot penetrate (smaller than about approximately 4 to 6 Å diameter pore mouth).

3.1.3 Other definitions and terms used in this test method are defined in Terminology D 3766.

3.2 *Symbols*:

3.2.1 *For Mercury Intrusion*:

W = weight of sample,
W_c = weight of sealed empty sample cell,
W'_C = weight of sealed sample cell filled with mercury,
W_s = weight of sealed sample cell with sample,
W'_S = weight of sealed sample cell with sample filled with mercury,
V_{Hg}^C = volume of mercury in empty sample cell (volume of sample cell),
V_{Hg}^S = volume of mercury in cell with sample,
V_S^{Hg} = sample volume, cm^3,
V_{Hg} = specific sample volume,
V_P = particle volume,
D_P = particle density,
W_b = weight mercury reservoir after filling buret with sample.

3.2.2 *For Helium Pycnometry*:

V_C = volume of sample cell and associated tubing, cm^3,
V_R = reference volume, cm^3,
V_S^{He} = sample volume, cm^3,
V_{Cyl} = volume of calibration cylinder, cm^3,
V_{STD} = volume of calibration standard, cm^3,
V_{He} = specific sample volume,
P'_1 = pressure in empty sample cell, psig or Pascals,
P'_2 = pressure in empty sample cell, after the reference volume has been included in the system, psig or Pascals,
P_1 = pressure in sample cell with sample or calibration standard before the reference volume has been included in the system, psig or Pascals,
P_2 = pressure with sample or calibration standard in the sample cell, after the reference volume has been included in the system, psig or Pascals,
W_1 = tare weight of sample cup, g,
W_2 = weight of sample + tare weight of sample cup, g,
W_3 = weight of sample, g,
D_t = true density,
$P.V.$ = pore volume.

4. Summary of Test Method

4.1 The total pore volume of a catalyst or catalyst carrier is determined as the difference between the particle volume and the true volume, measured by mercury intrusion and helium

[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] *Annual Book of ASTM Standards*, Vol 05.05.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

pycnometry, respectively. The particle volume is determined by mercury intrusion at ambient pressure and the true volume is determined by helium displacement at pressures above ambient.

5. Significance and Use

5.1 This test method provides for the measurement of volume of pores that are in the range of catalytic importance and possibly for adsorption processes.

6. Apparatus

6.1 *For Mercury Intrusion*:

6.1.1 *Chamber,* capable of holding the sample cell (commonly referred to as a penetrometer), which contains the sample. This chamber must be capable of being evacuated and contain enough mercury to fill the penetrometer.

6.1.2 *Glass Sample Cell (penetrometer),* having a wide base and narrow bore stem. If the sample is powder, the penetrometer should have a provision in the base to prevent fine particles from passing into the stem when the cell is evacuated. The penetrometer must have the capability of being sealed.

6.1.3 *Vacuum Pump,* capable of attaining pressures of less than 0.05 torr.

6.1.4 *Valve,* for choosing vacuum and vent, for evacuation of the sample cell and filling the sample cell, respectively.

6.1.5 *Valve,* for rapid evacuation or venting of the system.

6.1.6 *Valve,* for controlled evacuation or venting.

6.1.7 A method or device to prevent mercury vapor from being vented into the room through the vacuum pump and to prevent contaminants from entering the vacuum pump.

6.1.8 *Pressure-Measuring Device,* capable of reading in the range 0 to 1000 torr or higher.

6.1.9 *Balance,* measuring to the nearest 1 mg (±0.001 g).

6.2 *For Mercury Intrusion with a Burette*—A schematic diagram of the burette is shown in Fig. 1. It has the following features:

6.2.1 *Glass Sample Cell,* with a needle valve suitable for handling mercury. The tip, which is submerged in the mercury reservoir, should be narrow enough so as to prevent drops of mercury from becoming lost if the reservoir is removed for weighing.

6.2.2 *Burette,* a calibrated narrow bore tube ending in a curved tip in the sample cell to prevent fine particles from passing into the burette. There is a clear mark on the burette at 23 cm above the curved tip.

6.2.3 *Manifold,* with a splash bulb and appropriate needle valves for choosing either vacuum or vent.

6.2.4 *Mercury Reservoir with Lid,* capable of containing the amount of mercury necessary to fill the sample cell and burette while the tip of the sample cell valve is still submerged in mercury. A weighing bottle of 5 cm diameter and 3 cm height is suitable.

6.2.5 *Vacuum Pump,* capable of attaining pressures of 0.05 torr.

6.2.6 *Cold Trap,* to prevent mercury vapor from being vented into the laboratory and to prevent contaminants from entering the vacuum pump.

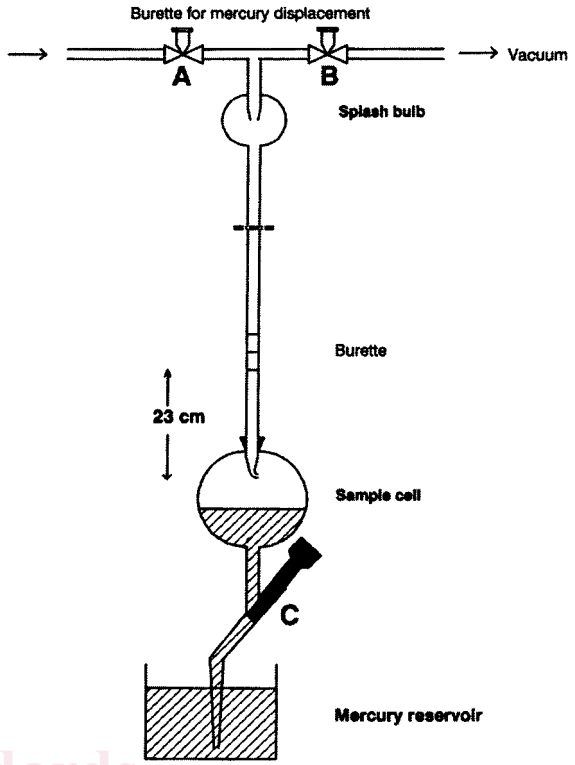

FIG. 1 Schematic Diagram of Burette

6.3 *For Helium Pycnometry*—A schematic diagram of the pycnometer apparatus is shown in Fig. 2. It should be constructed from metal and have the following features:

6.3.1 *Sample Cell,* having a volume suitable for the desired sample size and calibrated to the nearest 0.1 cm^3. This volume is indicated in Fig. 2.

6.3.2 *Reference Volume (V_R),* a precisely calibrated volume known to the nearest 0.02 cm^3.

6.3.3 *Pressure Transducer,* (0 to 25 psig or 0 to 172.3 kPa) with minimum volume displacement and linear within 0.1 %.

6.3.4 *Pressure Relief Valve,* set to 25 psig (172.3 kPa), to avoid overpressurization of the transducer.

6.3.5 *Filter,* to prevent powder from contaminating the pressure transducer.

6.3.6 *Input Flow Control Valves,* to control pressurization.

6.3.7 *Output Flow Control Valves,* to vent the gas.

6.3.8 *Valve,* to connect the reference volume to the sample cell.

6.3.9 *Non-Porous Calibration Standard,* (preferably stainless steel) of known volume which fills ¼ to ⅔ of the sample cup.

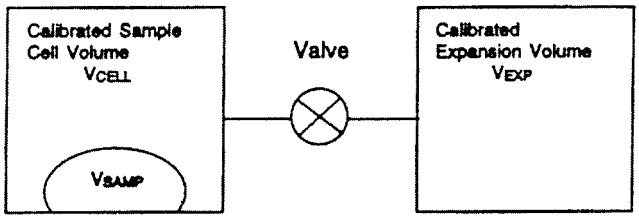

FIG. 2 Pycnometer Apparatus