



Standard Test Method for Determination of Nitrogen Adsorption and Desorption Isotherms of Catalysts and Catalyst Carriers by Static Volumetric Measurements¹

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1. Scope

1.1 This test method covers the determination of nitrogen adsorption and desorption isotherms of catalysts and catalyst carriers at the boiling point of liquid nitrogen.² A static volumetric measuring system is used to obtain sufficient equilibrium adsorption points on each branch of the isotherm to adequately define the adsorption and desorption branches of the isotherm. Thirty points evenly spread over the isotherm is considered to be the minimum number of points that will adequately define the isotherm.

1.2 The values stated in SI units are to be regarded as the 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:³

- D 3663 Test Method for Surface Area of Catalysts
- D 3766 Terminology Relating to Catalysts and Catalysis
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E 456 Terminology Relating to Quality and Statistics
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—See Terminology D 3766.

3.2 *Symbols*:

- PH_1 = initial helium pressure, torr.
- PH_2 = helium pressure after equilibration, torr.
- TH_1 = temperature of manifold at initial helium pressure, °C.
- TH_2 = temperature of manifold after equilibration, °C.
- P_1 = initial N_2 pressure, torr.
- T_1 = manifold temperature at initial N_2 pressure, K.
- T'_1 = manifold temperature at initial N_2 pressure, °C.
- P_2 = pressure after equilibration, torr.
- T_2 = manifold temperature after equilibrium, K.
- T'_2 = manifold temperature after equilibrium, °C.
- P_3 = initial N_2 pressure during desorption, torr.
- T_3 = manifold temperature at initial N_2 pressure, K.
- T'_3 = manifold temperature at initial N_2 pressure, °C.
- P_4 = pressure after equilibration during desorption, torr.
- T_4 = manifold temperature after equilibration, K.
- T'_4 = manifold temperature after equilibration, °C.
- P_0 = liquid nitrogen vapor pressure, torr.
- T_s = liquid nitrogen temperature, K.
- X = relative pressure, $P_{2(4)}/P_0$.
- V_d = volume of manifold, cm^3 .
- V_s = the dead-space volume factor, cm^3 (STP)/torr.
- W_s = mass of sample, g.
- W_1 = tare of sample tube, g.
- W'_2 = sample mass + tare of tube after degassing, g.
- W_2 = sample mass + tare of tube after adsorption, g.
- V_{ds} = volume of nitrogen in the dead-space, cm^3 (STP).
- V_1 = see 12.4.3.
- V_2 = see 12.4.4.
- V_t = see 12.4.5.
- V_{ad} = see 12.4.7.
- V_{de} = see 12.5.

4. Summary of Test Method

4.1 The sample is heated and evacuated to remove adsorbed vapors from the surface. The nitrogen adsorption branch of the isotherm is determined by evacuating the sample, cooling the sample to the boiling point of liquid nitrogen (~ 77.3 K), and subsequently adding stepwise, known amounts of nitrogen gas to the sample in such amounts that the form of the adsorption isotherm is adequately defined and the saturation pressure of nitrogen is reached. Each additional dose of nitrogen is

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² Adamson, A. W., *Physical Chemistry of Surfaces*, 3rd ed., John Wiley & Sons, New York, NY, 1976, p. 532.

³ 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.