



National Institute of Standards & Technology

Report of Investigation

Surface Area Reference Materials 8570-8572

8570 Kaolin
8571 Alumina
8572 Silica-Alumina

Prepared by the American Society for Testing and Materials (ASTM)
Distributed by the National Institute of Standards and Technology (NIST)

These Reference Materials (RMs) consist of 25 g of the respective powders and are intended for use in calibrating instruments and evaluating the reliability of analytical methods used for determining the Brunauer, Emmet, and Teller (BET) surface area of powders and catalysts.

RMs 8570-8572 were prepared and analyzed through a cooperative program between NIST and ASTM Committee D32 (Subcommittee D32.01 on Physical-Chemical Properties). The cooperating laboratories used ASTM Standard Test Methods D 3663-84 and D 4567-86 [1,2].

Static (Volumetric) Surface Area Determination (D 3663-84): Test method D 3663-84 [1] covers the determination of surface areas of catalysts that have Type II or IV nitrogen adsorption isotherms, and at least 1 m²/g of area. A volumetric measuring system is used to obtain at least four data points which fit on the linear BET line [3]. The surface area of the catalyst is determined by measuring the volume of nitrogen gas adsorbed at various low-pressure levels by the catalyst sample. Pressure differentials caused by introducing the catalyst surface area to a fixed volume of nitrogen in the test apparatus are measured and used to calculate BET surface area. The results of the round robin test (24 participants) are:

Reference Material	Mean Specific Surface Area (m ² /g)
8570 Kaolin	10.9
8571 Alumina	158
8572 Silica Alumina	291

Supporting data and statistical analyses of within-laboratory repeatability and between-laboratory reproducibility are presented in the accompanying ASTM Research Report RR: D32-1006.

CAUTION: The sample pretreatment is critical in order to obtain the reported value. These measurements were done with sample pretreatment consisting of heating to about 300 °C (573 K) and degassing the sample at that temperature for a minimum of 3 h, at a pressure not to exceed 10⁻³ torr, as prescribed in paragraphs 7.8-7.9 in D3663-84 [1].

Revision of this certificate was coordinated through the Standard Reference Materials Program by J.C. Colbert.

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Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

Single-Point, Continuous Flow Surface Area Determination (D 4567-86): Test method D 4567-86 [2] covers the single-point determination of the surface area of catalysts and catalyst carriers that exhibit Type II or Type IV nitrogen adsorption isotherms using a nitrogen-helium flowing gas mixture. This test method is applicable for the determination of total surface areas from 0.1 to 300 m², where rapid surface area determinations are desired [1]. Since the single-point calibration uses an approximation of the BET equation, the multi-point BET method (D 3663) is preferred to the single-point method. The results obtained in the round robin study by the continuous flow method are:

Reference Material	Specific Surface Area (m ² /g)
8570 Kaolin	10.3
8571 Alumina	153
8572 Silica-Alumina	277

Supporting data and statistical analyses of within-laboratory repeatability and between-laboratory reproducibility are presented in the accompanying ASTM Research Report RR: D32-1019.

CAUTION: The sample pretreatment is critical and must be followed as prescribed in D 4567-86 in order to reproduce the above value: namely, the sample must be heated to 300 °C (573 K) and degassed at the temperature for a minimum of 1 h (see paragraphs 9.6-9.7 of D 4567-86).

REFERENCES

- [1] ASTM Standard Test Method for Surface Area of Catalysts, D 3663-84, Annual Book of ASTM Standards, Vol. 5.03, 1986.
- [2] ASTM Standard Test Method of Single-Point Determination of Specific Surface Area of Catalysts using Nitrogen Adsorption by Continuous Flow Method, D 4567-86, Annual Book of ASTM Standards, Vol. 5.03, 1986.
- [3] Brunauer, Emmet, and Teller, J. Am. Chem. Soc., No. 60:309, 1938.