Designation: C 1069 – 86 (Reapproved 2004)ε1

Standard Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption[1]

This standard is issued under the fixed designation C 1069; 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.

ε[1] NOTE—The address in footnote 3 was changed in May 2004.

1. Scope

1.1 This test method covers the determination of the specific surface area of aluminas and silicas used in the manufacture of ceramics. The test method is a general one, permitting the use of any modern commercial nitrogen adsorption apparatus but strictly defining the outgassing procedure. Calculations are based on the Brunauer-Emmett-Teller (BET) equation.

1.2 *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:* [2]

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals

3. Summary

3.1 An appropriate size sample is degassed for 1 h at 423 K before proceeding with the analysis as prescribed by the manufacturer of the instrument used. The sample is accurately weighed after analysis.

3.2 Calculations are based on the BET equations, adapted where necessary for the equipment being used. The cross-sectional area of the nitrogen molecule is taken as 0.162 nm^2.

4. Significance and Use

4.1 Both sellers and purchasers of alumina and quartz will find the test method useful to determine the specific surface area and indirectly as a measure of the particle size for material specifications, manufacturing control, and research and development.

5. Apparatus

5.1 *Specific Surface Area Analyzer*, recently (after 1970) manufactured commercial equipment employing low-temperature (77 K) nitrogen adsorption. This test method may be dynamic or static.

5.2 *Degassing Equipment*, suitable to the instrument used.

5.3 *Analytical Balance*, having a sensitivity of 1.0 mg.

6. Reagents and Materials

6.1 *Liquid Nitrogen.*

6.2 Cylinder of compressed nitrogen gas, high purity as specified by manufacturer, with pressure regulator.

6.3 Cylinder of compressed helium gas, high purity as specified by manufacturer, with pressure regulator, for static instruments or for dynamic instruments having gas mixing ability and employing the multipoint procedure or,

6.4 Cylinder of compressed, approximately 0.30 mol fraction, nitrogen in helium with pressure regulator, for other dynamic instruments employing the single point procedure.

7. Procedure

7.1 *Sample Preparation*—Blend the gross sample very well. For non-free-flowing materials mechanical agitation is necessary (for example, V-type blender with intensifier). If sample splitting of free-flowing materials is necessary, use a spinning riffler.[3]

7.2 *Sample Degassing*

7.2.1 Sample size is related to the equipment being used and shall conform to the equipment manufacturer's recommendations for highest precision.

7.2.2 The degassing technique (for example, vacuum, no-vacuum) shall conform to the equipment manufacturer's recommendations.

7.2.3 The degassing temperature shall be 1 h at 423 K.

7.2.4 Samples must be analyzed immediately after degassing.

[1] This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Prodcuts and is the direct responsibility of Subcommittee C21.04 on Raw Materials.

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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] Spinning rifflers manufactured by Quantachrome Corp., 1900 Corporate Drive, Boynton Beach, FL 33426 or by Gilson Company, Inc., P.O. Box 200, Lewis Center, OH 43035-0200, which has been found suitable for this purpose.

7.3 *Analysis*—The analysis procedure recommended by the manufacturer as their most precise shall be followed.

8. Calculations

8.1 The procedure recommended by the manufacturer as being the most complete shall be followed.

9. Report

9.1 Report the following information:

9.1.1 *Results*—In units of m^2/g.

9.1.2 Specific surface area as the average of two determinations, each made on separately degassed samples. If the results do not agree within 5 % the procedure should be reviewed, the equipment checked, and the test repeated a third time.

9.1.3 From a practical standpoint it is recommended that the following guidelines be used in reporting data:

9.1.3.1 From 1 to 99 m^2/g—no more than two significant figures.

9.1.3.2 From 100 to 200 m^2/g—to the nearest 1 m^2/g.

9.1.3.3 From 200 m^2/g and above—to the nearest 5 m^2/g.

TABLE 1 Analyses of Variance

Surface Area, m^2/g[A]	Repeatability Interval, R_1, m^2/g	Reproducibility Interval, R_2, m^2/g
1.1	0.05	0.19
1.3	0.12	0.26
8.4	0.40	2.07
9.3	0.44	0.86
25	0.63	5.2
101	4.8	11.0
205	8.2	33.2

[A] Averages from round-robin study.

10. Precision and Bias

10.1 *Precision*—Six laboratories cooperated in a testing program involving various materials having specific surface areas ranging from 1 to 200 m^2/g. The determinations were made in duplicate on separate samples and days. Practice E 173 was used to obtain the analyses of variance in Table 1.

10.2 *Bias*—No absolute method for surface area determinations is recognized and, therefore, it is not possible to discuss bias of results obtained.

11. Keywords

11.1 alumina; nitrogen adsorption; particle size; quartz