INTERNATIONAL

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# Standard Test Method for Precipitated Silica-Surface Area by Multipoint BET Nitrogen Adsorption[1]

This standard is issued under the fixed designation D 1993; 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 a procedure which is used to measure the surface area of precipitated hydrated silicas by the conventional Brunauer, Emmett, and Teller (BET)[2] theory of multilayer gas adsorption behavior using multipoint determinations, similar to that used for carbon black in Test Methods D 4820. This test method specifies the sample preparation and treatment, instrument calibrations, required accuracy and precision of experimental data, and calculations of the surface area results from the obtained data.

1.2 This test method is used to determine the nitrogen surface area of precipitated silicas with specific surface areas in the range of 1 to 50 $hm^2/kg$ (10 to 500 $m^2/g$).

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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.* The minimum safety equipment should include protective gloves, sturdy eye and face protection, and means to deal safely with accidental mercury spills.

## 2. Referenced Documents

2.1- *ASTM Standards:*

D 1799 Practice for Carbon Black—Sampling Packaged Shipments[3]
D 1900 Practice for Carbon Black—Sampling Bulk Shipments[3]
D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[3]
D 4820 Test Methods for Carbon Black—Surface Area by Multipoint B.E.T. Nitrogen Adsorption[4]

## 3. Significance and Use

3.1 This test method is used to measure the surface area of precipitated, hydrated silicas that is available to the nitrogen molecule using the multipoint (B. E. T.) method.

3.2 Solids adsorb nitrogen and, under specific conditions, the adsorbed molecules approach a monomolecular layer. The quantity in this hypothetical monomolecular layer is calculated using the BET equation. Combining this with the area occupied by the nitrogen molecule yields the total surface area of the solid.

3.3 This test method measures the estimated quantity of nitrogen in the monomolecular layer by adsorption at liquid nitrogen temperature and at several (at least five) partial pressures of nitrogen.

3.4 Before a surface area determination can be made it is necessary that the silica be stripped of any material which may already be adsorbed on the surface. The stripping of adsorbed foreign material ~~(by heating under vacuum)~~ eliminates two potential errors. The first error is associated with the weight of the foreign material. The second error is associated with the surface area that the foreign material occupies.

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[1] This test method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

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[2] Brunauer, Emmett, and Teller, *Journal of the American Chemical Society*, Vol 60, 1938, p. 309.

[3] *Annual Book of ASTM Standards*, Vol. 09.01.

~~[4] Two commercial automated vacuum instruments found satisfactory are (*a*) Model ASAP 2400 Accelerated Surface Area and Porosimetry Instrument, available from Micromeritics Instrument Corporation, 800 Goshen Springs Road, Norcross, GA 30071, and, (*b*) Autosorb Automated Gas Adsorption System, available from Quantachrome Corporation, 5 Aerial Way, Syosset, NY 11791.~~

[4] Discontinued—see *1999 Annual Book of ASTM Standards* , Vol. 09.01.

## 4. Apparatus

~~4.1 *Classical Glass Vacuum Apparatus*, or equivalent constructed as shown in Fig. 1, including diffusion and vacuum pumps, manometer, gas buret, gas reservoir, and sample cell similar to that illustrated in Fig. 1.~~

~~4.2 Optionally, commercial~~

4.1 Commercial instruments are available[5] for the measurement of nitrogen surface area by the multipoint BET method. These ~~are automated versions~~ may be of the ~~vacuum apparatus described herein.~~ "flowing gas" or the "vacuum-volumetric" type.

NOTE 1—Automated ~~vacuum~~ instruments will provide results equivalent to the procedure described herein if careful calibration of the instrument, equivalent sample preparation, adherence to manufacturer's instruction for instrument operation, and equivalent data handling and calculations are performed.

4.~~3~~2 *Sample Cells*~~, which, when attached to the vacuum system, will maintain pressure below 1.3 mPa (10 nm Hg).~~

~~4.4 For automated instruments: sample tubes, filler rods, stoppers, isothermal jacket (if required in manufacturer's procedure),~~ and ~~sample filler tube or powder funnel to fit~~ other peripheral equipment as recommended by the ~~neck of~~ manufacturer for the ~~sample tube.~~

~~4.5 *Dewar Flasks*, 1-L size (furnished with commercial instruments).~~

~~4.6 *McCleod Gage*, or equivalent means to measure pressure to less than 1 mPa (8 nm Hg).~~

~~4.7~~ instrument used.

4.3 *Balance, Analytical*, with 0.1-mg sensitivity.

4.~~8~~4 *Heating Mantles*, or ~~equivalent,~~ other sample preparation station, capable of maintaining a temperature of 160 ± 5°C on the sample.

## 5. Reagents

5.1 ~~*Purity of Water*—Unless otherwise indicated, references to water (and ice prepared from it) shall be understood to mean distilled water or water of equal purity (for gas vacuum rack, Fig. 1).~~

~~5.2~~ *Liquid Nitrogen*.

5.2 *Nitrogen Gas*, cylinder, or other source of prepurified nitrogen gas as specified by the manufacturer of the instrument.

5.3 ~~*Ultra-High Purity Nitrogen Gas*, cylinder, or other source of prepurified nitrogen gas.~~

~~5.4 *Ultra-High Purity Helium*~~*Helium Gas*, cylinder, or other source of prepurified helium ~~gas.~~

~~5.5 *Instrument Grade Mercury* (for gas vacuum rack, Fig. 1).~~ gas, as specified by the manufacturer of the instrument.

## 6. Standard Reference Silicas

6.1 *None Required*— This test method is used to determine surface area of candidate silicas. Reference silicas are available[6] for checking agreement with data obtained in the interlaboratory test used in preparation of this test method.

## 7. Sampling

7.1 No separate practice for sampling silicas is available. However, samples may be taken in accordance with Test Methods D 1799 or D 1900, whichever is appropriate.

## 8. ~~Preparation of Apparatus~~

### ~~*Classical Vacuum Apparatus*~~

~~NOTE 2—This procedure only need be performed for the initial calibration of the gas buret or when a modification is made to the gas buret.~~

~~8.1 Zero the manometer by evacuating both arms of the manometer to a pressure below 1.3 mPa (10 nm Hg) and adjusting the height of the mercury columns to the same zero reading in both arms.~~

~~8.2 *Determine Gas Buret Constant*:~~

~~8.2.1 Install a sample cell of approximately 30 cm$^3$ volume on the sample cell position of the apparatus, assuring no vacuum leaks.~~

~~8.2.2 Evacuate the sample cell, manometer, and gas buret to a pressure below 1.3 mPa (10 nm Hg).~~

~~8.2.3 Immerse the sample cell in a water-ice bath contained in a Dewar flask such that the entire sample bulb and neck are covered.~~

~~8.2.4 Fill the gas buret to approximately 50 % of its capacity with helium.~~

~~8.2.5 Obtain initial gas buret level and temperature—$V_{B1}$, $T_{B1}$ (*K*).~~

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~~[5] Precipitated silica samples are available~~

[5] Commercial automated instruments found satisfactory may be obtained from ~~Forcoven Products, Inc., 123 Martin~~ Micromeritics Instrument Corporation, One Micromeritics Drive, ~~Porter, TX 77365. Samples are available in three surface areas: A, 13.8; B, 5.7;~~ Norcross, GA 30093–1877, website: www.micromeritics.com, and ~~C, 16.8 hm$^2$/kg.~~ Quantachrome Instruments, 1900 Corporate Drive, Boynton Beach, FL 33426, website: www.quantachrome.com.

[6] Precipitated silica samples are available from Forcoven Products, Inc., 123 Martin Drive, Porter, TX 77365. Samples are available in three surface areas: A, 13.8; B, 5.7; and C, 16.8 hm$^2$/kg.

~~8.2.6 Transfer approximately 2 cm$^3$ of helium to the sample cell.~~

~~8.2.7 Read and record the gas buret level and temperature after helium dosing—$V_{B2}$, $T_{B2}$, ($K$).~~

~~8.2.8 Read and record the pressure in the sample cell after dosing ($P_c$); and the sample cell temperature, that is, ice-bath temperature $T_c$($K$).~~

~~8.2.9 Repeat 8.2.5-8.2.8 at least two times to obtain a total of three data sets.~~

~~8.2.10 Remove the sample cell and fill the sample cell with approximately 25 cm$^3$ of instrument grade mercury. Record the mass of the mercury used to ±0.001 g, $W_m$.~~

~~8.2.11 Reinstall the sample cell containing the mercury onto the apparatus.~~

~~8.2.12 Re-evacuate the sample cell, manometer and gas buret to a pressure below 1.3 mPa (10 nm Hg).~~

~~8.2.13 Repeat 8.2.3-8.2.9.~~

~~8.3 *Buret Factor Calculations*:~~

~~8.3.1 For each set of data (see 8.2.9) determine the pressure change by measuring the difference in buret readings before and after helium dosing as follows:~~

~~$P_{B1} = V_{B1} - V_{B2}$~~ ~~(1)~~

~~8.3.2 Determine the total buret pressure difference for each data set as follows:~~

~~$P_{BT} = (V_{B1} - V_{B2})_1 + (V_{B1} - V_{B2})_2 + ...$~~ ~~(2)~~

~~8.3.3 Determine buret pressure to cell pressure ratio for each data set as follows:~~

~~$A = P_{BT}/P_C$~~ ~~(3)~~

~~8.3.4 Determine the ratio of cell temperature to buret temperature for each data set as follows:~~

~~$B = T_C/T_B$~~ ~~(4)~~

~~8.3.5 Determine the product of the temperature and pressure for each data set as follows:~~

~~$C = A \times B$~~ ~~(5)~~

~~8.3.6 Determine the average $C$ value for the empty sample cell ($C_E$) and the cell with the mercury ($C_m$).~~

~~8.3.7 Determine the volume of mercury used as follows:~~

~~$V = W_m$/density of mercury~~
~~density of mercury~~
~~$= 13.5955$ Mg/m$^3$ at 0°$C$~~ ~~(6)~~

~~8.3.8 Determine the buret volume factor corrected to standard temperature and pressure (STP) as follows:~~

~~$F_B = [V/(C_E - C_m)][273.15/101.32]$~~ ~~(7)~~

~~*Preparation of Automated Vacuum Instruments*~~

~~8.4 Calibrate manifold temperature sensor, in accordance with manufacturer's procedure (monthly).~~

~~8.5 Zero master pressure gage, in accordance with manufacturer's procedure (daily).~~

~~8.6 Calibrate system volume, in accordance with manufacturer's procedure (calibrate after servicing valves, transducers, or any part of instrument plumbing).~~

## ~~9.~~ Sample Preparation Procedure

### ~~*Classical Suction Apparatus*~~*Automated Instrument*

~~9~~8.1 Weigh (to 0.1 mg) a ~~clean and dry~~ clean, dry, degassed sample ~~cell (with its~~ tube with stopper ~~in place) to the nearest 0.1 mg~~ and filler rod if required. Record the mass.

~~9.2 Transfer into the cell~~

8.2 Transfer a sample of ~~the~~ silica to be tested so that the ~~cell~~ sample tube contains approximately 50 m$^2$ of silica surface area (see Note ~~3~~ 2 and ~~Note 3 and 9.2.1). Insert glass stopper in sample tube.~~ 8.2.1).

NOTE ~~3~~2—If the silica sample contains more than about 6 % moisture, it may be dried at 110°C to 2 to 6 % moisture. A very dry silica (less than 1 % moisture) is difficult to transfer due to static charge buildup.

~~9~~8.2.1 If the surface area of the silica is unknown, assume a surface area of 7.5 hm$^2$/kg and weigh out approximately 0.5 g of sample.

~~9.3 With apparatus at atmospheric pressure, attach~~

8.3 Place the sample ~~cell (with sample) to the suction apparatus.~~

~~9.4 Turn on the mechanical vacuum pump. After pressure is reduced below 130 Pa (1 mm Hg), proceed to 9.5.~~

~~9.5 Start the diffusion pump.~~

~~9.6 Place a heating mantle around the sample tube and adjust the temperature so that the sample experiences 160°C. Degas for 1 h or longer as required to obtain and hold a pressure less than 1.3 mPa (10 nm Hg); record the temperature and heating time.~~

~~9.7 Remove the heating mantle and allow sample and sample cell to cool to room temperature.~~

~~*Automated Suction Instrument*~~

~~9.8 Weigh (to 0.1 mg) a clean, dry, degassed sample tube with filler rod and stopper and record the mass.~~

~~9.9 Use sample filler tube or powder funnel to transfer a sample of silica to be tested so that the sample tube contains approximately 50 m$^2$ of silica surface area (see Note 3 and 9.2.1).~~

~~9.10 Attach the sample~~ assembly (with sample) ~~to~~ at the degas ~~port.~~

~~9.11 Set~~ station. Degas the ~~degas suction meter set point to 100 millitorr. Start degas procedure~~ sample in accordance with manufacturer's instructions.

~~9.11.1 The~~

8.3.1 The silica must be completely degassed. While samples at normal moisture and moderate surface area are completely degassed in 1 h at 160°C, the inability to hold pressure (in a vacuum-type instrument), moisture condensation in the cold part of the sample cell, or poor reproducibility are indications that longer degassing times may be required. *Do not change the degassing temperature.*

~~9.12 Place a~~

8.4 Set the heating ~~mantle around sample, set~~ for the desired temperature of 160°C, and ~~start heating~~ degas in accordance with manufacturer's ~~procedure. See 9.11.1.~~

procedure.

NOTE ~~4~~3—To obtain 160°C sample temperature, a higher temperature on the ~~heating mantle~~ heater may be necessary. The ~~mantle~~ heater temperature and set point necessary may be determined by way of a temperature sensor in the sample, for example, a thermometer, during a trial run.

~~9.13 Remove heating mantle~~

8.5 Remove from heat and allow sample and sample tube to cool to room temperature. If moisture is present at the tube neck after 1 h, abort run and pre-dry sample at 110°C in accordance with Note ~~3~~ 2 and repeat sample preparation procedure. When cool, ~~backfill to atmospheric pressure with helium,~~ remove the sample tube from the degas port in accordance with manufacturer's procedure, stopper, weigh, and record the mass to 0.1 mg. Calculate degas sample weight using weight from ~~9.8~~ 8.1 as tare. The degassed sample weight is inserted into program of calculations.

## ~~10. Calibration Procedure (Classical Suction Apparatus)~~

~~NOTE 5—This procedure is designed to measure the dead volume (that is, helium factor) in the system using helium, as helium is not adsorbed onto the surface of the silica at the temperature of liquid nitrogen.~~

~~10.1 When the sample has cooled, immerse the sample bulb to a depth of 10 cm in the liquid nitrogen contained in the Dewar flask.~~

~~10.1.1 Since liquid nitrogen tends to evaporate rather rapidly, be sure to keep the level constant for the duration of the test.~~

~~10.2 Measure the helium factor of the sample cell system (that is, calibrate the system) using helium gas as follows:~~

~~10.2.1 Admit a dose of helium to the gas buret that is approximately 50 % of the volume of the buret.~~

~~10.2.2 Read and record gas buret pressure ($P_{B1}$) and temperature ($T_B$).~~

~~10.2.3 Dose sample cell with approximately 2 cm$^3$ of helium. Read and record manometer pressure ($P$).~~

~~10.2.4 Read and record gas buret pressure ($P_{B2}$), having dosed the sample with helium.~~

~~10.2.5 Repeat incremental dosing of sample cell with helium twice more; read and record buret pressure, manometer pressure, and buret temperature for each dosing.~~

~~10.2.6 Calculate helium factor in accordance with 12.2.~~

~~10.2.6.1 When helium factors are calculated, the three results should not differ by more than ±0.004 cm$^3$/Pa (±0.0005 cm$^3$/mm Hg). If their differences are more than this, repeat 10.2.1 to 10.2.5 to obtain factors that are within 0.004 cm$^3$/Pa (0.0005 cm$^3$/mm Hg) of each other.~~

~~10.3 While cell is still immersed in liquid nitrogen, evacuate helium from the entire system. After approximately 15 min, read the pressure with the McCleod or equivalent gage. When a pressure less than 1.3 mPa (10 nm Hg) has been attained, begin measurement procedure.~~

## ~~11.~~

## 9. Measurement Procedure

~~*Classical Suction Apparatus*~~

~~11.1 Fill gas buret with nitrogen gas to approximately 75 % of its volume.~~

~~11.2 Dose the sample cell with an amount of nitrogen gas to give an approximate pressure reading of 4.7 kPa (35 mm Hg). This is a partial pressure ($P/P_o$) of ≈0.05. Read and record buret pressures ($P_{B1}$, $P_{B2}$), as done in 10.2, and temperature ($T_B$) along with the manometer pressure ($P$) for each dose.~~

~~11.3 Allow the pressure in the sample cell-manometer system to equilibrate for 0.5 h or more to obtain a constant pressure. Read and record manometer pressure ($P$).~~

~~11.4 For subsequent doses, introduce 1 to 1.5 cm$^3$ of nitrogen to the sample in successive doses. Obtain a minimum of five data sets in the pressure range of 6 kPa (45 mm Hg) to 22 kPa (160 mm Hg), or partial pressure ($P/P_o$) of 0.05 to 0.20. In determining silica surface areas, it is not necessary to take readings at manometer pressures higher than 22 kPa (160 mm Hg) since this value is beyond the linear region of the adsorption isotherm.~~

~~11.5 Obtain the saturation vapor pressure of liquid nitrogen ($P_o$) by accurately measuring the barometric pressure and adding 2 kPa (15 mm Hg) to its value as follows:~~

~~$P_o$ = barometric pressure (kPa) + 2 kPa (8)~~

~~11.5.1 In principle, a more accurate means of measuring $P_o$ is to immerse an identical, empty reference cell in a Dewar flask to a depth equivalent to the sample cell. Admit nitrogen gas into the reference cell to obtain equilibrium pressure ($P_o$), that is approximately 103 kPa (775 mm Hg).~~

~~11.6 With both suction pumps running, open the stopcocks so that the sample cell is open to the manifold. Remove the liquid nitrogen from around the sample cell, allowing adsorbed nitrogen to exhaust into the suction line.~~

~~11.7 Warm the sample and sample cell to room temperature, close the valve between the sample cell and the suction manifold and readmit nitrogen to bring the pressure back to atmospheric pressure in the sample tube.~~

~~11.8 Dry the sample tube with an absorbent cloth or paper, remove the sample tube from the vacuum apparatus, and seal with its own stopper.~~

~~11.9 Weigh the closed sample cell and sample; record their combined mass to 0.1 mg.~~

*~~Automated Suction~~Automated Instruments*

~~11.10 For~~

9.1 For automated system insert prepared tube containing sample into isothermal jacket (if called for in manufacturer's procedure), install on analysis port, and insert run conditions/report options into computer program as required.

~~11.11 For~~

9.2 For automated system when partial pressures are requested, select 0.05 and 0.2 and three points between 0.05 and 0.2. Begin run.

~~11.11.1 Be~~

9.2.1 Be sure to input degassed weight of sample obtained in ~~9.13.~~

~~11.12 When~~ 8.5.

9.3 When measurements are complete and sample tube has warmed to room temperature, dry the sample tube, remove it from the instrument, and seal it with its stopper.

~~11.13 Weigh (to 0.1 mg) the closed sample tube and sample, and record their combined mass.~~

## ~~12.~~

## 10. Calculations

10.1 For automated instruments, software automatically calculates results for ~~Classical Suction Apparatus~~

~~12.1 *Sample Mass:*~~ the chosen reports.

~~Mass of sample (dried) = (mass of cell + sample from 11.9) − (mass of cell from 9.1)~~

~~12.2 *Helium Factor*—Calculate helium factor as follows:~~

~~Helium factor = $\Sigma [1/P_n][(273.15V_B)$~~
~~$(P_{B2} - P_{B1})]/101.32T_B$ (10)~~

~~where:~~
~~$V_B$ = volume of buret (see Section 10),~~
~~$T_B$ = temperature of buret in Kelvin ($K$ = 273.15 + °C),~~
~~$P_{B1}$ = initial pressure in buret,~~
~~$P_{B2}$ = final pressure in buret,~~
~~$P$ = pressure in manometer, and~~
~~$n$ = number of data sets.~~

~~12.3 *Nitrogen Surface Area*:~~

~~12.3.1 Calculate volume of nitrogen admitted to the nearest ± 0.0001 cm$^3$ as follows:~~

~~$V_{ADM} = F_B \times (P_{B2} - P_{B1})/T_B$ (11)~~

~~where:~~
~~$V_{ADM}$ = volume of gas admitted at STP, cm$^3$,~~
~~$F_B$ = buret factor,~~

~~$P_{B2}$ = final pressure in buret (read after 0.5 h of equilibration),~~
~~$P_{B1}$ = initial pressure in buret (read before each dose), and~~
~~$T_B$ = buret temperature (temperature of insulating water jacket).~~

~~12.3.2 Calculate volume of nitrogen not adsorbed to the nearest ± 0.0001 cm$^3$ as follows:~~

~~$V_{NA} = P \times \text{helium factor} \times \text{gas law correction}$ (12)~~

~~where:~~
~~$V_{NA}$ = volume not adsorbed, cm$^3$,~~
~~$P$ = manometer pressure, and gas law correction = (1 + deviation) = 1.0472 + 0.0064P.~~

~~12.3.2.1 Deviation is calculated from the formula:~~

~~$\text{Deviation} = 0.0472 + 0.0064P$ (13)~~

~~12.3.3 Calculate volume of nitrogen adsorbed to the nearest 0.0001 cm$^3$ as follows:~~

~~$V_{ADS} = V_{ADM} - V_{NA}$ (14)~~

~~where:~~
~~$V_{ADS}$ = volume of nitrogen adsorbed each dose, cm$^3$,~~
~~$V_{ADM}$ = volume admitted at STP, cm$^3$, and~~
~~$V_{NA}$ = volume not adsorbed, cm$^3$.~~

~~12.3.4 Calculate total volume of nitrogen adsorbed per gram of specimen to the nearest 0.0001 cm$^3$/g as follows:~~

~~$V_{ADS/g} = \frac{V_{ADS}}{\text{sample mass}}$ (15)~~

~~where:~~
~~$V_{ADS/g}$ = total volume of nitrogen adsorbed per gram of silica, cm$^3$/g.~~

~~12.3.5 Determine the surface area of the silica using a plot from the Brunauer, Emmet, and Teller (BET) equation as follows:~~

~~$\frac{P}{V_{ADS/g}(P_o - P)} = 1/V_mC + C - 1/V_mC \times P/P_o$ (16)~~

~~12.3.5.1 Plot $P/P_o$ on $X$-axis versus $P/[V_{ADS/g}(P_o - P)]$ on $Y$-axis, for data sets having $P/P_o$ in the range of 0.06 to 0.20 (linear region of BET equation).~~

~~where:~~
~~$P$ = manometer pressure, kPa,~~
~~$V_{ADS/g}$ = total volume of nitrogen adsorbed per gram of silica, cm$^3$/g,~~
~~$P_o$ = saturation vapor pressure of nitrogen (kPa),~~
~~$V_M$ = volume of nitrogen per gram that covers one monomolecular layer, cm$^3$/g,~~
~~$C$ = a constant, that is a function of average heat of adsorption of the monomolecular layer,~~
~~$B$ = Y-axis intercept, ± 0.00001,~~
~~$M$ = slope of the straight line (determine slope to ±0.00001), and~~
~~$V_M$ = $1/_{B + M}$~~

NOTE ~~6—Standard linear regression calculations can also be used to determine~~ 4—If the ~~slope and intercept of this function.~~

~~12.3.6 Calculate~~ correlation coefficient calculated for the ~~nitrogen surface area~~ data analysis is low, see Test Methods D 4820 for a methodology to improve the ~~nearest 0.01 hm$^2$/kg as follows:~~

~~$\text{Surface area (hm}^2\text{/kg)} = V_m \times 4.35 \times 0.1$ (17)~~

~~where:~~
~~4.35 = area (m$^2$) occupied~~ correlation by ~~1 cm$^3$ of nitrogen =~~
~~$(6.02 \times 1023)\ (16.2 \times 10{-}20)/_{22\ 400}$~~
~~$6.02 \times 10^{23}$ = Avogadro's number, molecules per mole,~~
~~$16.2 \times 10^{-20}$ = area of nitrogen molecule, m$^2$, and~~
~~22 400 = number of cubic centimetres occupied by~~ discarding one ~~mole of gas at STP.~~

~~12.4 For automated instruments, software automatically calculates results for the chosen reports using the weight obtained in 9.13.~~

## ~~13.~~ or more points.

## 11. Report

1~~3~~1.1 Report the following information:

1~~3~~1.1.1 Proper sample identification.

1~~3~~1.1.2 Number of data points used to obtain the results.

1~~3~~1.1.3 The nitrogen surface area of the sample reported to the nearest 0.01 $hm^2/kg$.

## 1~~4~~2. Precision and Bias

1~~4~~2.1 This precision and bias section has been prepared in accordance with Practice D 4483, which should be referred to for terminology and other statistical calculation details.

1~~4~~2.2 A Type 1 interlaboratory precision was evaluated in April 1990. Both repeatability and reproducibility are short-term. Duplicate determinations were made on each of the test silicas on each of two days, a few days apart. A test result, as specified by this test method, is obtained on one measurement of the surface area.

1~~4~~2.3 Three different precipitated silicas were used, representing low (less than 10.0 $hm^2/kg$), medium (10.0 to 16.0 $hm^2/kg$) and high (greater than 16.0 $hm^2/kg$). These were tested in five laboratories. The tests included the classical vacuum rack (one laboratory) and the automatic instruments (four laboratories).

1~~4~~2.4 The results of the precision calculations for repeatability and reproducibility are given in Table 1 for each of the silicas evaluated.

1~~4~~2.5 Repeatability, *r*, and reproducibility, *R*, vary over the range of surface areas ~~measured (Fig. 2).~~

~~14.6~~ measured.

12.6 *Repeatability*— The repeatability, *r*, of this test method has been established as the appropriate value tabulated in Table 1. Two single test results, obtained with this test method on the same instrument and with the same operator, that differ by more than this tabulated *r* (for any given mean surface area) must be considered as derived from different or nonidentical sample populations.

1~~4~~2.7 *Reproducibility*— The reproducibility, *R*, of this test method has been established as the appropriate value tabulated in Table 1. Two single test results obtained with this test method, in two different laboratories, that differ by more than the tabulated *R* (for any given mean surface area) must be considered as derived from different or nonidentical sample populations.

1~~4~~2.8 Repeatability and reproducibility expressed as a percent of the mean surface area, (*r*) and (*R*), have equivalent application statements as above for *r* and *R*. For the ( *r*) and (*R*) statements, the difference in the two single test results is expressed as a percent of the arithmetic mean of the two test results.

1~~4~~2.9 *Bias*—In test method terminology, bias is the difference between an average surface area and the reference (or true) surface area. Reference surface areas do not exist for this test method since the surface area is exclusively defined by this test method. Bias, therefore, cannot be determined.

## 1~~5~~3. Keywords

1~~5~~3.1 nitrogen adsorption surface area; precipitated hydrated silica; silicas; surface area

**TABLE 1 Type 1 Precision of Surface Area of Silica**

| Silica | Mean, $hm^2/kg$ | Within Laboratory | | | Between Laboratories | | |
|---|---|---|---|---|---|---|---|
| | | *Sr*, $hm^2/kg$ | *r*, $hm^2/kg$ | (*r*), | *Sr*, $hm^2/kg$ | *R*, $hm^2/kg$ | (*R*), |
| A | 13.78 | 0.141 | 0.399 | 2.9 | 0.148 | 0.419 | 3.0 |
| B | 5.67 | 0.079 | 0.223 | 3.9 | 0.094 | 0.267 | 4.71 |
| C | 16.78 | 0.201 | 0.569 | 3.4 | 0.377 | 1.067 | 6.4 |

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