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Standard Test Method for
Advanced Ceramic Specific Surface Area by Physical Adsorption[1]

This standard is issued under the fixed designation C1274; 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 determination of surface area of advanced ceramic materials. This test method specifies general procedures that are applicable to many commercial physical adsorption instruments. This test method provides specific sample outgassing procedures for listed materials, including silicon carbide, silicon nitride, and zirconium oxide. It includes additional general outgassing instructions for other advanced ceramic materials. The multipoint equation of Brunauer, Emmett and Teller*~~

1.1 This test method covers the determination of the surface area of advanced ceramic materials (in a solid form) based on multilayer physisorption of gas in accordance with the method of Brunauer, Emmett, and Teller (BET) **(1)**[2] ~~(BET) along with the single point approximation of the BET equation form the basis for all calculations.~~

~~1.2 This test method does not include all existing procedures appropriate for outgassing advanced ceramic materials. The included procedures provided acceptable results for samples analyzed during round robin testing. The investigator must determine the appropriateness of listed procedures.~~

~~1.3 This test method uses SI units as standard. State all numerical values in terms of SI units unless specific instrumentation software reports surface area using alternate units. In this case, present both reported and equivalent SI units in the final written report. Many instruments report surface area as m~~ and based on IUPAC Recommendations (1984 and 1994) **(2)** and **(3)**. This test method specifies general procedures that are applicable to many commercial physical adsorption instruments. This test method provides specific sample outgassing procedures for selected common ceramic materials, including: amorphous and crystalline silicas, TiO_2 ~~/g, instead of using correct SI units (m~~. kaolin, silicon nitride, silicon carbide, zirconium oxide, etc. The multipoint BET **(1)** equation along with the single point approximation of the BET equation are the basis for all calculations. This test method is appropriate for measuring surface areas of advanced ceramic powders down to at least 0.05 m^2 ~~/kg).~~ (if in addition to nitrogen, krypton at 77.35 K is utilized as an adsorptive).

1.2 This test method does not include all existing procedures appropriate for outgassing of advanced ceramic materials. However, it provides a comprehensive summary of procedures recommended in the literature for selected types of ceramic materials. The investigator shall determine the appropriateness of listed procedures.

1.3 The values stated in SI units are to be regarded as standard. State all numerical values in terms of SI units unless specific instrumentation software reports surface area using alternate units. In this case, provide both reported and equivalent SI units in the final written report. It is commonly accepted and customary (in physical adsorption and related fields) to report the (specific) surface area of solids as m^2/g, and, as a convention, many instruments (as well as certificates of reference materials) report surface area as m^2 g^{-1}, instead of using SI units (m^2 kg^{-1}).

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]
D1993 Test Method for Precipitated Silica-Surface Area by Multipoint BET Nitrogen Adsorption
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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*A Summary of Changes section appears at the end of this standard.

2.2 *ISO Standards:*[4]

ISO 9277 Determination of specific surface area of solids by gas adsorption using the BET method

ISO 15901-2:2006 Pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption - Part 2 Analysis of mesopores and macropores by gas adsorption

ISO 8213:1986 Chemical products for industrial use - Sampling techniques-Solid chemical products in the form of particles varying from powders to coarse lumps

ISO 18757 Fine ceramics (advanced ceramics, advanced technical ceramics) – Determination of specific surface area of ceramic powders by gas adsorption using the BET method

3. Terminology

3.1 *Definitions:* [5]

3.1.1 *adsorbate*, *n*—material that has been retained by the process of adsorption.

3.1.2 *adsorbent*, *n*—any solid having the ability to concentrate significant quantities of other substances on its surface.

3.1.3 *adsorption*, *n*—~~a process~~—process in which ~~fluid~~ molecules are concentrated on a surface by chemical or physical forces, or both.

3.1.4 *adsorption isotherm*, *n*—relation between the quantity of adsorbate and the equilibrium (relative) pressure of the adsorptive, at constant temperature.

3.1.4.1 *Discussion*—Typically, the amount adsorbed is presented on an isotherm as volume in cm^3 STP (Standard Temperature and Pressure, that is, 273.15 K and 101325.02 Pa) normalized per mass of sample.

3.1.5 *adsorptive*, *n*—any substance available for adsorption.

~~3.1.5 aliquant~~

3.1.6 *aliquot*, *n*—a representative portion of a whole that divides the whole leaving a remainder.

~~3.1.6~~

3.1.7 *molecular cross-sectional area*, *n*—molecular area of the adsorbate, that is, the area occupied by an adsorbate molecule in the completed closed-packed monolayer.

3.1.8 *monolayer capacity*, *n*—amount of the adsorbate (expressed as number of moles, volume at STP, or weight) that forms a closed-packed (complete) monomolecular layer over the surface of the adsorbent.

3.1.9 *outgassing*, *n*—~~the evolution~~—evolution of gas from a material ~~in~~under a vacuum or inert gas ~~flow,~~flow at or above ambient temperature.

~~3.1.7~~3.1.10 *physical adsorption (van der Waals adsorption)*, *n*—the binding of an adsorbate to the surface of a solid by forces whose energy levels approximate those of condensation.

~~3.1.8~~

3.1.11 *relative pressure*, *n*—ratio of the equilibrium adsorption pressure, *p*, to the saturation vapor pressure, p_O.

3.1.12 *saturation vapor pressure*, *n*—vapor pressure of the bulk liquefied adsorbate at the temperature of adsorption.

3.1.13 *surface area*, *n*—~~the total~~—total surface area of the surface of a powder or solid including both external and accessible internal surfaces (from voids, cracks, open porosity, and fissures).

3.1.13.1 *Discussion*—The surface area may be calculated by the BET (~~Brunauer, Emmett, and Teller~~[2]~~equation~~ **(1)** ~~equation~~ from gas adsorption data obtained under specific conditions. It is useful to express this value as the specific surface ~~area, for example,~~ area (see 3.1.13), that is, surface area per unit ~~weight~~mass of sample (m^2 /~~g).~~ ~~3.1.9~~g^{-1}).

3.1.14 *surface area (BET)*, *n*—~~the total~~—total surface area of a solid calculated by the BET (~~Brunauer, Emmett, Teller~~[2]equation, **(1)** ~~equation,~~ from ~~nitrogen~~gas adsorption or desorption data obtained under specific conditions.

~~3.1.10~~3.1.15 *surface area, specific (SSA)*, *n*—~~the area,~~—area, per unit mass of a granular or powdered or formed porous solid, of all external ~~plus~~and internal surfaces that are accessible to a penetrating gas or liquid.

4. Summary of Test Method

~~4.1 An appropriate sized sample (to provide at least the minimum surface area required for reliable results for the instrument or apparatus used) is outgassed under appropriate conditions prior to analysis.~~

~~4.2 (Multipoint BET Analyses Only)—Volume of gas adsorbed, or desorbed, is determined for a minimum of four relative pressures within the linear BET transformation range of the physical adsorption, or desorption, isotherm characteristic of the advanced ceramic. The linear range is that which results in a least square correlation coefficient of 0.999 (preferably 0.9999) or greater for the linear relationship used in the BET graph (1/(adsorbed volume * (1/relative pressure — 1))). Typically, the linear range includes relative pressures between 0.05 and 0.30, however, microporous materials usually require use of a range of lower relative pressures, such as 0.01 to 0.10.~~

4.1 An appropriately sized (to provide at least the minimum surface area required for reliable results; refer to requirements provided by the manufacturer of the instrument or apparatus being used) aliquot of sample is outgassed under appropriate conditions prior to analysis. For details on outgassing methods and examples of specific outgassing conditions recommended for selected ceramic materials, see Section 11.

4.2 The adsorptive gas is admitted to a sample container held at a constant temperature. The amounts adsorbed are measured in equilibrium with the adsorptive gas pressure, p, and plotted against the relative pressure, p/p_0, (where p_0 is the saturation vapor pressure) to give an adsorption isotherm. Adsorption isotherms may be obtained by volumetric (manometric) measurements or by the carrier gas flow measurements (flow volumetric method) and gravimetric techniques. This test method employs volumetric and flow volumetric methods.

4.3 *(Multipoint BET Analyses Only)*—The volume of gas adsorbed, or desorbed, is determined for a minimum of four relative pressures within the linear BET transformation range of the physical adsorption, or desorption, isotherm characteristic of the advanced ceramic. The linear range is that which results in a least square correlation coefficient of 0.995 (preferably 0.999) or greater for the linear relationship (see linear form of BET equation, in Annex A1). Typically, the linear range includes relative pressures between 0.05 and 0.30 **(5) (6)**. However, microporous materials usually require use of a range of lower relative pressures (often a linear BET range can be found in the relative pressure range from 0.01 to 0.1 **(6) (7)**. For details, see Annex A2.

4.4 *(Single Point BET Analyses Only)*—~~Volume of gas adsorbed, or desorbed, is determined at the highest known relative pressure within the linear BET transformation range of the physical adsorption, or desorption, isotherm. Typically, a relative pressure of 0.30 is used. (It may be necessary to perform a multipoint analysis of the material first to determine the optimum single point relative pressure.)~~

~~4.4The sample is accurately weighed (to at least 1% of the sample mass) after analysis. It is important to use an analytical balance to determine the sample weight. The physical adsorption instrument or apparatus measures the total amount of gas adsorbed onto, of desorbed from, the sample under analysis. The sample weight is then used to normalize the measured results. Any error in the sample weight will be propagated into the final BET surface area.~~

~~4.5Calculations are based on the BET equation, as required by the instrument being used for the determination. The cross sectional area for the adsorbate is taken to be 0.162 nm~~—The volume of gas adsorbed, or desorbed, is determined at the highest known relative pressure within the linear BET transformation range of the physical adsorption, or desorption, isotherm. Typically, a relative pressure of 0.30 is used. However, it may be necessary to perform a multipoint analysis of the material first to determine the optimum single point relative pressure.

4.5 The physical adsorption instrument or apparatus measures the total amount of gas adsorbed onto, or desorbed from, the sample under analysis. The sample mass is then used to normalize the measured results. Therefore, it is important to use an analytical balance to determine the sample weight. The mass of dry and outgassed sample, recorded to the nearest 0.1 mg, shall be used. Any error in the sample weight will be propagated into the final BET surface area result.

4.6 Typical steps involved in the evaluation of the BET surface area (see Annex A1 for calculation details):

4.6.1 Transformation of a physisorption isotherm into the BET plot,

4.6.2 An assessment of the monolayer capacity (multi-point or single-point method). (See Eq A1.1-A1.6 in Annex A1.), and

Note 1—Monolayer capacity can be expressed alternatively in terms of STP volume (V^m), weight (w_m), or number of moles (n_m), of adsorbate in a complete monolayer per 1 g of sample.

4.6.3 Calculation of the specific surface area (SSA), a_s (see Eq A1.7 in Annex A1), which requires knowledge of the monolayer capacity as well as the effective molecular cross-sectional area of the adsorbate. Recommended customary values for molecules of N_2 ~~if nitrogen is used as the adsorptive. Use the appropriate value recommended by the instrument manufacturer for adsorptives other than nitrogen. Report this cross sectional area with the BET surface area results.~~ at 77.35 K, Ar at 87.27 K, and Kr at 77.35 K, are provided in Table 1.

5. Significance and Use

~~5.1Both suppliers and users of advanced ceramics can benefit from knowledge of the surface area of these materials. Results of many intermediate and final processing steps are controlled by, or related to, specific surface area of the advanced ceramic.~~

5.1 Advanced ceramic powders and porous ceramic bodies often have a very fine particulate morphology and structure that are marked by high surface-to-volume (S-V) ratios. These ceramics with high S-V ratios commonly exhibit enhanced chemical reactivity and lower sintering temperatures. Results of many intermediate and final ceramic processing steps are controlled by, or related to, the specific surface area of the advanced ceramic. The functionality of ceramic adsorbents, separation filters and membranes, catalysts, chromatographic carriers, coatings, and pigments often depends on the amount and distribution of the porosity and its resulting effect on the specific surface area.

5.2 This test method determines the specific surface area of advanced ceramic powders and porous bodies. Both suppliers and users of advanced ceramics can use knowledge of the surface area of these ceramics for material development and comparison, product characterization, design data, quality control, and engineering/ production specifications.

6. Interferences

6.1 This test method can be used to determine the internal and external surface of a powder or solid only after these surfaces have been cleaned of any physically adsorbed molecules. Such adsorbed species, for ~~example,~~example water or volatile organic

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TABLE 1 ~~P~~Cross-Sec~~tis~~ti~~o~~n ~~foal~~ Areas of ~~Singele Poinct~~ an~~ed~~
~~Multip~~ Co~~i~~mmon~~t~~ BET M~~ly~~ U~~sethod~~ Adsorptives

			Precision Single Point BET Method
~~Material~~	~~Number of Laboratories~~	~~Surface Area (average)~~	Repeatability Standard Deviatio n S_r
Adsorptive	Temperature, K	~~Recommended Value of Cross-Se~~)	Repe~~a~~ctional Area, nm^2
~~Reproducibility Standard Deviation S_R~~	~~Repeatability Limit~~	~~Reproducibility Limit~~	~~Repeatability C.V.~~	~~Reprod~~
Nitrogen	77.35	~~Reproducibility Limit~~	~~Repeatability C~~0.V.	~~Reprodue~~
~~A1N~~	~~6~~	~~2.82~~	0.09	
~~Si3N4 (SNT)~~	~~17~~	~~9.30~~	0.24	
~~Si3N4 (SNR)~~	~~6~~	~~12.55~~	0.79	
~~SiC~~	~~16~~	~~13.27~~	0.25	
~~Y2O3-ZrO2~~	~~9~~	~~17.72~~	0.34	

~~This table was calculated using the relationship: limit = 1.96 × √2 × standard deviation~~

			Precision Multi Point BET Method
~~Material~~	~~Number of Laboratories~~	~~Surface Area (average)~~	Repeatability Standard Deviation S_r
Argon	77.35	~~Surface Area (average)~~	Repeatability Standard Deviation $S_{r0.138}{}^B$
~~Reproducibility Standard Deviation S_R~~	~~Repeatability Limit~~	~~Reproducibility Limit~~	0.142
Argon	87.27	~~Reproducibility Limit~~	0.142
~~Repeatability C.V.~~	~~Reproducibility C.V.~~	~~77.V.~~	
Krypton			
~~A1N~~	6	~~2.83~~	0.15
~~A1N35~~	6	~~2.83~~	0.15
~~Si3N4 (SNT)~~	~~11~~	~~10.06~~	0.41
~~Si~~^{C}~~+0~~	~~14.22~~	~~0.28~~	1.50
~~Y2O3-ZrO2~~	~~6~~	~~18.46~~	0.43

~~This table was calculated using the relationship: limit = 1.96 × √2 × standard deviation~~

[A]Very often the orientation of the adsorbed N_2 molecules (having quadruple moment) can be affected by specific interactions with polar groups on the surface of adsorbent (i.e. in case of highly polar surfaces, such as with hydroxylated oxide surface groups). **(8-10)** This can lead to a significant reduction in the effective cross-sectional area. If the standard value for N_2 molecule (0.162 nm^2 at 77.35 K) is used, the BET surface area of hydroxylated silica surfaces can be overestimated by 20%. Therefore, in case of ceramics with surfaces of high polarity, argon (which is a chemically inert monoatomic gas) adsorption at 87.3 K is an alternative adsorptive recommended for surface area determination, since the cross-sectional area of argon (0.142 nm^2 at 87.27 K) is less sensitive to differences in structure of the adsorbent surface.

[B]The use of argon at 77.35 K (which is approximately 6.5 K below the triple point of bulk argon) is considered to be less reliable than nitrogen, because at 77.35 K the structure of the argon monolayer may be highly dependent on the surface chemistry of the adsorbent. The cross-sectional area for argon at 77.35 K is not well defined. The value of 0.138 nm^2, as given in the table is based on the assumption of a closed-packed liquid monolayer, and can also be considered to be the customary value.

[C]The use of krypton at 77.35 K allows to manometrically measure very low uptakes with acceptable accuracy. However, similar to argon at 77.35 K, krypton at 77.35 K is significantly below the triple point temperature of bulk krypton (approximately 38.5 K), and the structure of the krypton monolayer may be strongly affected by the surface chemistry of the adsorbent. This will directly influence the effective krypton cross-sectional area. The value given in the table can be considered to be a customary value.

compounds, ~~prevent~~affect physical adsorption of the gas probe molecules used to measure surface area. Therefore, it is necessary to remove these adsorbed contaminants prior to surface area analysis. Generally, such ~~outgassing~~procedure is performed by evacuating or ~~flushing~~purging the sample with inert gas. Outgassing can be accelerated by using elevated temperatures, provided no irreversible sample changes occur. Typical minimum vacuum levels attained are 10^{-1} Pa. ~~Typical flushing~~ Commonly used purging gases are helium, nitrogen, or a mixture of the two. ~~O~~The outgassing procedure is optimal or complete, or both when: *(1)*

duplicate surface area analyses produce results within expected instrument repeatability limits, ~~when a~~ *(2)* constant residual vapor pressure is maintained upon isolation from the vacuum source, or ~~when flushing~~ *(3)* purging gas composition is unaffected while passing over the sample.

6.2 The outgassing procedures and temperatures shall not produce any changes in composition, phase, or surface morphology of the powder specimens. The outgas temperature limits are determined by the stability limits of the powder samples.

7. Apparatus

7.1 ~~*Classical Vacuum Apparatus*—Refer to Test Method~~ Manometric (Volumetric) Apparatus—See Test Method D1993 ~~for apparatus description.~~ and ISO 15901-2 for description of technology.

7.2 *Automated and Dynamic Flow Instruments*—~~Commercial instruments are available from several manufacturers for the measurement of specific surface area by physical adsorption. Some are automated versions of the classical vacuum apparatus. Others may use a gravimetric technique to determine the amount of adsorbed gas on the sample surface. Additionally, commercial instruments are available which measure physical adsorption based on the dynamic flow method.~~ —Commercial instruments are available from several manufacturers for the measurement of specific surface area by physical adsorption. Some are automated versions of the classical vacuum apparatus. Others may use a gravimetric technique to determine the amount of adsorbed gas on the sample surface. Additionally, commercial instruments are available that measure physical adsorption based on the dynamic flow method.

7.3 *Sample Cells*, that when attached to the adsorption apparatus will maintain isolation from the atmosphere equivalent to a specified (helium) leak rate determined by the manufacturer of the instrument.

7.4 *Heating Mantle or Equivalent*, capable of maintaining a temperature in range from 100 to 300 ± 10 °C.

7.5 *Analytical Balance*, with 0.1 mg sensitivity.

7.6 *Oven (Optional)*, gravity convection, capable of maintaining a temperature of 115 ± 10 °C.

8. Reagents and Materials

8.1 *Liquid Nitrogen*.

8.2 ~~*Nitrogen*, 99.99 mole percent, with the sum of O~~Ultra-High Purity Nitrogen, 99.99 mol %, with the sum of O_2, Ar, CO_2, hydrocarbons (as CH_4), and H_2O totaling less than 10 ppm, dry and oil-free, ~~cylinder,~~ from a cylinder or other source of purified nitrogen.

8.3 ~~*Helium*, 99.99 mole percent, with the sum of N~~Ultra-High Purity Helium, 99.99 mol %, with the sum of N_2, O_2, Ar, CO_2, hydrocarbons (as CH_4), and H_2O totaling less than 10 ppm, dry and oil-free, ~~cylinder,~~ from a cylinder or other source of purified helium, if needed for determination of void space above sample.

8.4 ~~*Blended Nitrogen and Helium*, dry and oil-free, cylinder, or other source of blended gases. The actual composition of the blend must be known. For use with dynamic flow instruments only.~~ Ultra-High Purity Blended Nitrogen and Helium, dry and oil-free, from a cylinder or other source of blended gases. The actual composition of the blend shall be known. For use with dynamic flow instruments only.

8.5 *Liquid Argon*.

8.6 *Ultra-High Purity Argon*, 99.99 mol %, with the sum of O_2, Ar, CO_2, hydrocarbons (as CH_4), and H_2O totaling less than 10 ppm, dry and oil-free, from a cylinder or other source of purified argon.

8.7 *Ultra-High Purity Krypton*, 99.99 mol %, with the sum of O_2, Ar, CO_2, hydrocarbons (as CH_4), and H_2O totaling less than 10 ppm, dry and oil-free, from a cylinder or other source of purified argon.

8.8 *Comments on Proper Selection of Adsorbate*:

8.8.1 Nitrogen at its boiling point (about 77.35 K) is the most commonly used adsorptive for customary ceramic materials (customary cross-sectional area of 0.162 nm^2). (See Table 1).

8.8.2 In case of ceramic materials with very low specific surface, the sensitivity of the instrument when using nitrogen adsorption may be insufficient. Therefore, for the BET surface area analysis of ceramics with specific surface areas below 1 m^2 g^{-1}, it is recommended to use krypton adsorption at liquid nitrogen temperature. Application of Kr at 77.35 K as adsorptive allows for volumetric measurement of very low uptakes with acceptable accuracy (as a consequence of the low p_0 of about 2.63 torr (360.63 Pa)) and for assessing specific surface areas down to at least 0.05 m^2. For krypton at 77.35 K, the number of molecules trapped in the void volume of the sample cell is significantly reduced (to 1/300th) compared to the conditions of nitrogen adsorption at the same temperature.

8.8.3 The results of measurements with different adsorptives may deviate from each other because of different molecular areas, different accessibilities to pores, different measuring temperatures, and so forth.

9. Sampling, Test Specimens, and Test Units

~~9.1 No specific instructions are given. However, it is important that the aliquant being analyzed represent the larger bulk sample from which it is taken. The bulk sample should be homogenized before any sampling takes place. Best results are obtained when a flowing bulk material is temporarily diverted into a collector for an appropriate time. It is better to sample the entire flow for a short time than to sample a portion of the flow for a longer time. Collecting several small aliquants and combining them improves the reliability of the sampling process. Rotating rifflers are available that satisfy these requirements.~~ Hazards

9.1 Precautions applying to the use of LN_2, LAr, compressed gasses, and specific properties of powder samples shall be observed.

10. Calibration and Standardization

~~10.1 *Classical Vacuum Apparatus*—Refer to Test Method~~ Sampling, Test Specimens, and Test Units

10.1 The goal of powder sampling is to collect a small amount of powder from the bulk quantity such that this smaller fraction is representative of the characteristics of the entire bulk. No mandatory instructions are given. However, it is advisable to perform statistical sampling in accordance with ISO 8213:1986 since it is important that the aliquot being analyzed represent the larger bulk sample from which it is taken. Homogenize the bulk sample before any sampling takes place. Best results are obtained when a flowing bulk material is temporarily diverted into a collector for an appropriate time. It is better to sample the entire flow for a short time than to sample a portion of the flow for a longer time. Collecting several small aliquots and combining them improves the reliability of the sampling process. Rotating rifflers that satisfy these requirements are commercially available.

10.2 Obtain enough powder samples to run one or more analyses in accordance with the equipment sample size recommendations.

11. Sample Preparation Procedure

11.1 Remove physically adsorbed impurities from the sample surface prior to the determination of an adsorption isotherm. This can be achieved at elevated temperatures under vacuum or by inert gas flow. It is crucial to outgas the sample under conditions (that is, time and temperature), which provide a clean sample surface at the beginning of the analysis while avoiding irreversible changes to the surface (solid sample shall not be altered). Most ceramic materials can be safely outgassed at relatively high temperatures (200 to 300°C) in a few hours, however, outgassing overnight at temperatures around 150°C is generally a safe way to achieve a clean sample surface and is commonly practiced (specific outgassing conditions recommended for selected ceramic materials are summarized in Table 2). Outgassing is complete when a steady value of the residual gas pressure, p_{out} (recommended 1 Pa or lower) of its composition, or when a steady sample mass, m, is reached.

11.2 If the ceramic material is wet (that is, when any moisture condensation is observed on the walls of the sample cell during sample outgassing), the sample requires pretreatment to remove excess moisture. Dry an aliquot of a ceramic sample at 90 to 110°C for 1 to 2 h. If the ceramic material is known to be substantially free of moisture, or subsequent preparation steps are known to be adequate for moisture removal, then this step may be omitted.

11.3 The following examples and conditions shall be carefully considered for any unknown ceramic material:

11.3.1 In most cases physisorbed water has to be removed from the adsorbent surfaces prior to the adsorption experiment. If the sample contains a large amount of water, either physisorbed on the surface, condensed in pores, or within its crystal structure, a special heating program is often needed that allows for a slow removal of most of the pre-adsorbed water at temperatures below 100°C, accompanied by a stepwise increase in temperature until the final outgassing temperature is reached. This is done to avoid potential structural damage of the sample due to surface tension effects and hydrothermal alteration. For outgassing of extremely sensitive samples and fine powders, so-called pressure controlled heating under vacuum is recommended. This outgassing procedure consists of heating steps achieved by controlling the temperature rise based on the gas pressure evolved from a porous material under vacuum. When, during evacuation and heating, a fixed pressure limit (usually approximately 7 to 10 Pa) is overtaken due to species/moisture evolved from the sample surface, the temperature ramp is stopped (the temperature is kept constant while evacuation is continued). When the pressure falls back below the limit, the temperature ramp is continued, etc., until a desired dwell temperature is achieved.

11.3.2 Take particular care during outgassing of certain ceramic materials (such as green-bodies) that may contain organic binders or other additives (see also ISO 18757). In such cases, any decomposition during outgassing at elevated temperatures could adversely change the chemical or physical properties, or both, of the solid (such as altered adsorption interactions with the analysis gas, certain pores or cracks blocked and thus inaccessible, particles sintered, crystalline phase transition, etc.). Preliminary thermal gravimetric analysis can be used to determine a proper outgassing temperature. In general, the surface area of a sample goes through a maximum as a function of temperature and it is recommended to use the highest value as the true surface area of the sample.

TABLE 2 Specific Examples of Outgassing Conditions (Under Vacuum) for Various Ceramic Materials

Ceramic material	Outgassing temperature T, °C	Outgassing time t, minimum, h
Silica	200 (or 150)	3 (or 12)
Quartz	200	3
Boehmite	105	1
Silica/alumina	300	3
Alumina	300	3
Silicon carbide	200	3
Silicon nitride	200	3
Kaolin (Kaolinite)	200	3
Titania	140	12
Glass fibers	120	24
Zirconium oxide	140	12