



Standard Test Method for Metal Powder Specific Surface Area by Physical Adsorption¹

This standard is issued under the fixed designation B922; 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 metal powders. The test method specifies general procedures that are applicable to many commercial physical adsorption instruments. The method provides specific sample outgassing procedures for listed materials. It includes additional general outgassing instructions for other metals. The multipoint equation of Brunauer, Emmett and Teller (BET),² along with the single point approximation of the BET equation, forms the basis for all calculations.

1.2 This test method does not include all existing procedures appropriate for outgassing metallic materials. The procedures included provided acceptable results for samples analyzed during interlaboratory testing. The investigator shall determine the appropriateness of listed procedures.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3.1 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^2/g , instead of using correct SI units (m^2/kg).

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

B215 Practices for Sampling Metal Powders

¹ This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.03 on Refractory Metal Powders.

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² Brunauer, S., Emmett, P. H., and Teller, E. "Adsorption of Gases in Multimolecular Layers." *Journal of the American Chemical Society*, Vol. 60, 1938, pp. 309-319.

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.

B243 Terminology of Powder Metallurgy

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 Definitions:

3.1.1 Refer to Terminology B243 for additional terms specific to metal powders.

3.2 Definitions of Terms Specific to This Standard:

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

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

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

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

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

3.2.6 *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.

3.2.7 *surface area, n*—the total area of the surface of a powder or solid including both external and accessible internal surfaces (from voids, cracks, open porosity, and fissures). The area may be calculated by the BET (Brunauer, Emmett, and Teller) equation from gas adsorption data obtained under specific conditions. It is useful to express this value as the specific surface area, for example, surface area per unit mass of sample (m^2/kg).

3.2.8 *surface area (BET), n*—the total surface area of a solid calculated by the BET (Brunauer, Emmett, Teller) equation, from nitrogen adsorption or desorption data obtained under specific conditions.

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

⁴ *ASTM Dictionary of Engineering, Science, and Technology*, 9th ed., ASTM International, West Conshohocken, PA, 2000.

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

4. Summary of Test Method

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

4.2 *Multipoint BET Analyses Only*—Volume of gas adsorbed, or desorbed, is determined as cm^3 corrected to standard temperature and pressure (STP) for a minimum of four relative pressures within the linear BET transformation range of the physical adsorption, or desorption, isotherm characteristic of the metal. The linear range is that which results in a least squares correlation coefficient of 0.9999 or greater for the relationship between BET transformation and relative pressure. Typically, the linear range includes relative pressures between 0.05 and 0.30.

4.3 *Single Point BET Analyses Only*—Volume of gas adsorbed, or desorbed, is determined as cm^3 corrected to standard temperature and pressure (STP) 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 first perform a multipoint analysis of the material to determine the optimum single point relative pressure.)

4.4 The sample is weighed to nearest 0.1 mg after analysis. It is important to use an analytical balance to determine the sample mass. The physical adsorption instrument 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 adsorption results. Any error in the sample mass will affect the final BET surface area.

4.5 Calculations 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^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.

5. Significance and Use

5.1 Both suppliers and users of metals 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 metal. The performance of many sintered or cast metal structures may be predicted from the specific surface area of the starting metal powder, or all or a portion of the finished piece.

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 water or volatile organic compounds, prevent 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 is performed by evacuating or flushing the sample. 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 gases are helium, nitrogen, or a mixture of the two. Outgassing is complete when duplicate surface area analyses produce results within expected instrument repeatability limits, when a constant residual vapor pressure is maintained upon isolation from the vacuum source, or when flushing gas composition is unaffected while passing over the sample.

7. Apparatus

7.1 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 make use of balanced adsorption technology. Additionally, commercial instruments are available which measure physical adsorption based on the dynamic flow method.

7.2 *Analytical Balance*, capable of weighing to the nearest 0.1 mg.

8. Reagents and Materials

8.1 *Liquid Nitrogen*.

8.2 *Nitrogen*, 99.999 mole percent, with the sum of O_2 , argon, CO_2 , hydrocarbons (as CH_4), and H_2O totaling less than 10 parts per million; dry and oil-free; cylinder, or other source of purified nitrogen.

8.3 *Helium*, 99.999 mole percent, with the sum of N_2 , O_2 , argon, CO_2 , hydrocarbons (as CH_4), and H_2O totaling less than 10 parts per million; dry and oil-free; 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.

9. Hazards

9.1 Precautions applying to the use of liquid nitrogen and compressed gases should be observed.

10. Sampling, Test Specimens, and Test Units

10.1 It is important that the test portion 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 riffles are available commercially which satisfy these sampling requirements. Refer to Practices B215 for information on the use of a chute sample splitter.

11. Calibration and Standardization

11.1 Follow manufacturer's instructions for calibration and operational verification of the instrument.

12. Outgassing

12.1 Weigh (to nearest 0.1 mg) a clean, empty sample tube, along with stopper or seal. Record the empty tube mass.

12.2 Add test portion to empty sample tube. Sample quantity should be sufficient to satisfy minimum surface area as required by manufacturer.

12.3 Attach prepared sample tube to outgassing port of instrument. Secure heating mantle or oven around sample tube at the time appropriate for sample.

12.4 Initiate outgassing program according to manufacturer's instructions. Program mantle or oven for initial outgassing temperature. Increase temperature as appropriate for the sample. Allow sample to continue to outgas until prescribed vacuum level or detector signal is achieved, or for prescribed outgassing time, or both. Samples analyzed during the interlaboratory study were heated for 2 h at 200°C.

12.5 Remove heating mantle or oven from sample tube. Allow sample tube to cool to ambient temperature. Remove and seal sample tube according to manufacturer's instructions.

12.6 Weigh sample tube (to nearest 0.1 mg) to obtain sample and tube mass. Record mass. Subtract empty sample tube mass determined in 12.1 to obtain outgassed sample mass. Record calculated mass.

13. Procedure

13.1 Attach appropriately prepared sample holder to analysis port according to manufacturer's instructions. Include any required hardware.

13.1.1 Use nitrogen as adsorptive for all tests. Use blended nitrogen and helium with dynamic flow instruments. Use pure nitrogen with volumetric instruments.

13.1.2 Use helium to determine sample holder void space with volumetric instruments as necessary.

13.1.3 Use liquid nitrogen as cryogen for all tests.

13.2 *Automated Instruments Only*—Select, or input, desired analysis and report parameters.

13.2.1 *Multipoint BET Analyses Only*—Use at least four analysis points in the linear BET transformation range of the isotherm characteristic of the sample. If necessary, input the outgassed sample mass. (The final mass should be determined and entered after the analysis.)

13.2.2 *Single Point BET Analyses Only*—Use highest relative pressure known to be in the linear BET transformation range of the isotherm. If necessary, input the outgassed sample mass. (The final mass should be determined and entered after the analysis.)

13.3 *Dynamic Flow Instruments Only*—Collect data points as volume of gas desorbed versus relative pressure.

13.3.1 *Multipoint BET Analyses Only*—Use at least four analysis points in the linear BET transformation range of the isotherm characteristic of the sample.

13.3.2 *Single Point BET Analyses Only*—Use highest relative pressure known to be in the linear BET transformation range of the isotherm.

13.4 Perform analysis using the specified conditions according to instrument manufacturer's instructions.

13.5 When the analysis has finished and the sample has warmed to room temperature, remove and seal the sample tube. Dry tube and weigh (to nearest 0.1 mg). Record the final tube and sample mass. Subtract the empty tube mass recorded in 12.1 to obtain the final sample mass. Record final sample mass.

13.6 *Automated Instruments Only*—Edit the file containing sample information to include the final sample mass. Generate final sample report.

14. Calculations

14.1 *Automated Instruments Only*—Software automatically calculates results for the chosen reports using the final mass input in 13.6.

14.2 *Dynamic Flow Instruments Only*—Follow manufacturer's instructions for multipoint, or single point, calculations. Use the final sample mass determined in 13.5 when calculating the specific surface area.

15. Report

15.1 Report the following information:

15.1.1 Complete sample identification.

15.1.2 Collected isotherm point(s) as volume adsorbed, or desorbed, versus relative pressure. Note whether adsorption or desorption isotherm is used. Note any units used other than standard.

15.1.3 Analysis gas used (with cross sectional area if other than nitrogen).

15.1.4 BET specific surface area. Note any units used other than standard.

15.1.5 Final sample mass. Note any units used other than standard.

15.1.6 Sample outgassing method, including total time and outgassing temperature(s).

16. Precision and Bias

16.1 An interlaboratory study is underway, conducted according to Practice E691. The study includes iron, tungsten, nickel, cobalt, molybdenum, chromium carbide, and tungsten carbide powders. Expected precision will be determined for these materials. No statement is given for other metal powders.

16.2 *Precision*—The repeatability standard deviation of single point specific surface area for one tungsten carbide sample has been determined to be $\pm 0.6\%$ relative standard deviation, based upon analyses in one laboratory, and for one nickel sample to be $\pm 1.4\%$ relative standard deviation. The reproducibility of this test method is being determined and will be available on or before June 30, 2011.

16.3 *Bias*—No information can be presented on the bias of the procedure in this test method for measuring specific surface area because no metal powder having an accepted reference value is available.

17. Keywords

17.1 BET surface area; metal powders; multipoint surface area; outgassing; physical adsorption; refractory metal powders; single point surface area; specific surface area; surface area

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B922 - 02(2010)) that may impact the use of this standard. (May 1, 2010)

(1) Subsection 16.2 has been updated to include a temporary precision statement to be used while the Interlaboratory Study is underway. Rationale: The repeatability of a single laboratory

will provide some guidance to the user of the Test Method as to what to expect from use of the procedures described here until a final precision statement can be completed.

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