Designation: B923 – 21

Standard Test Method for Metal Powder Skeletal Density by Helium or Nitrogen Pycnometry[1]

This standard is issued under the fixed designation B923; 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 skeletal density of metal powders. The test method specifies general procedures that are applicable to many commercial pycnometry instruments. The method provides specific sample outgassing procedures for listed materials. It includes additional general outgassing instructions for other metals. The ideal gas law forms the basis for all calculations.

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

1.3 *Units*—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 volume or density, or both, using alternative units. In this case, present both reported and equivalent SI units in the final written report. Many instruments report skeletal density as g/cm^3 instead of using correct SI units (kg/m^3).

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B215 Practices for Sampling Metal Powders

B243 Terminology of Powder Metallurgy

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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 definitions relating to metal powders.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *density, n*—the mass per unit volume of a material.

3.2.2 *density, skeletal, n*—the ratio of mass of discrete pieces of solid material to the sum of the volumes of the solid material in the pieces and closed pores within the pieces.

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

3.2.4 *skeletal volume, n*—the sum of the volumes of the solid material in the pieces and closed pores within the pieces.

4. Summary of Test Method

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

4.2 The sample is weighed to nearest 0.0001 g. It is important to use an analytical balance to determine the sample mass. The pycnometer measures the total displaced skeletal volume of the sample under analysis. The sample mass is then used to calculate the skeletal density of the metal. Any error in the sample mass will affect the calculated density. Some cleaning of the sample surface may take place inside the pycnometer.

[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] 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.

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

Therefore, it is best to reweigh the sample after analysis and use the final mass when calculating skeletal density.

4.3 Sample skeletal volume is determined a minimum of five times. Skeletal volume average and standard deviation are calculated using standard statistical methods.

4.4 Calculations are based on the ideal gas law, as required by the instrument being used for the determination. The assumption of ideal behavior is accepted as valid at analytical temperatures and pressures. For instruments designed with two pressure chambers, one a sample compartment, and the other a gas expansion chamber, the equation for sample volume calculation takes the form:

$$V_{sample} = V_{cell} - V_{exp} \cdot \left(\frac{P_2}{P_1 - P_2} \right) \quad (1)$$

where:

V_{sample} = calculated sample volume,
V_{cell} = calibrated sample compartment volume,
V_{exp} = calibrated expansion volume,
P_1 = measured gas pressure when only V_{cell} is filled with analysis gas, and
P_2 = measured gas pressure after expansion of the analysis gas into V_{exp}.

5. Significance and Use

5.1 Both suppliers and users of metals can benefit from knowledge of the skeletal density of these materials. Results of many intermediate and final processing steps are controlled by or related to skeletal density of the metal. In addition, the performance of many sintered or cast metal structures may be predicted from the skeletal density of the starting metal powder, for all or a portion of the finished piece.

6. Interferences

6.1 This test method can be used to determine the skeletal volume of a powder or solid only after the open pores have been emptied of any physically adsorbed molecules. Such adsorbed species (for example, water or volatile organic compounds) prevent entry of the gas probe molecules into the open porosity of the sample. Therefore, it is necessary to remove these adsorbed contaminants prior to pycnometry 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 those used for analysis. Outgassing is complete when duplicate skeletal volume analyses produce results within expected instrument repeatability limits. Some commercial instruments include capabilities for automated evacuation, or flushing of the sample, or both. Elevated temperatures should not be used when outgassing samples inside the pycnometer.

6.2 This test method can be used to determine the volume of a sample whose pores have been deliberately filled with a second phase. In this case, removal of the second phase should be avoided. Vacuum degassing or flushing of the sample is not necessary in this case.

7. Apparatus

7.1 Commercial instruments are available from several manufacturers for the measurement of skeletal volume by gas pycnometry. Some instruments perform calculations of skeletal volume, or density, or both, upon completion of the analysis. Others require manual calculation of skeletal volume and density.

7.2 *Analytical Balance*—A balance readable to 0.0001 g, with a capacity adequate for the mass of the test portion, and capable of determining the mass of the test portion to the nearest 0.001 g.

8. Reagents and Materials

8.1 *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.

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 *Other High Purity Gas,* dry and oil-free; cylinder, or other source of gas, if other gas is to be used as the analysis or flushing gas. The actual composition of the gas shall be known.

9. Hazards

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

10. Sampling, Test Specimens, and Test Units

10.1 No specific instructions are given. Nevertheless, 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 test portions and combining them improves the reliability of the sampling process. Rotating rifflers are available that satisfy these requirements. Refer to Practices B215 for information on the use of a chute sample splitter.

10.2 While there is no specific requirement for the test specimen size, result reliability increases as the percentage of the pycnometer sample capacity used increases. To this end, it is recommended that the test specimen have a skeletal volume greater than or equal to 10 % of the pycnometer sample capacity.

11. Calibration and Standardization

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

12. Conditioning

12.1 Weigh (to nearest 0.0001 g) a clean, empty sample holder. Record the empty sample holder mass.

12.2 Add sample aliquant to empty sample holder. Sample quantity should be sufficient to satisfy minimum skeletal volume as required by manufacturer. Weigh (to nearest 0.0001 g) and record sample and sample holder mass.

12.3 Sample outgassing may be performed inside the pycnometer. If so, proceed to the *Procedure* section of this test method. Otherwise, follow the remaining steps in this section for external outgassing.

12.3.1 Place prepared sample holder in outgassing device.

12.3.2 Program outgassing device for initial outgassing temperature. Increase temperature as appropriate for the sample. Allow sample to continue to outgas until prescribed vacuum level is achieved, or for prescribed outgassing time, or both.

12.3.3 The metal powders analyzed during the interlaboratory study were prepared inside the instruments by purging with analysis gas. Had preliminary outgassing been desired, a temperature of 200 °C applied for 1 h would have been used.

12.3.4 Reduce temperature of outgassing device to ambient. Remove sample holder.

12.3.5 Weigh sample holder (to nearest 0.0001 g) to obtain sample and sample holder mass. Record mass. Subtract empty sample holder mass determined in 12.1 to obtain outgassed sample mass. Record calculated mass.

13. Procedure

13.1 Place filled sample holder in pycnometer. Close sample chamber.

13.2 Use helium, nitrogen, or other high purity gas for analysis and flushing gas.

13.3 *Automated Instruments Only*—Select, or input, desired analysis and report parameters. Include outgassing parameters if sample preparation is performed as a part of the sample analysis. If necessary, input the outgassed sample mass. (The final mass should be determined and entered after the analysis.) Determine skeletal volume a minimum of five times.

13.4 *Manually Operated Instruments Only*—Collect five sets of analysis data according to manufacturer's recommended procedure for maximum accuracy and precision.

13.5 When the analysis has finished, remove the sample holder. Weigh holder (to nearest 0.0001 g). Record the final sample holder and sample mass. Subtract the empty sample holder mass recorded in 12.1 to obtain the final sample mass. Record final sample mass.

13.6 *Automated Instruments Only*—Input 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 *Manually Operated Instruments Only*—Calculate skeletal volume using collected data according to manufacturer's instructions. Use final sample mass from 13.5 to calculate skeletal densities. Calculated average and standard deviation for skeletal volume and density as described in Practice E691.

15. Report

15.1 Report the following information:

15.1.1 Complete sample identification.

15.1.2 Measured skeletal volumes and statistics. Note any units used other than standard.

15.1.3 Skeletal density determined. Note any units used other than standard.

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

15.1.5 Analysis gas used.

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

16. Precision and Bias

16.1 The precision of this test method is based on an interlaboratory study of Test Method B923 conducted in 2018. Each of 6 laboratories tested 9 different materials. Every "test result" represents an individual determination, and all participants reported triplicate test results. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:B09-1027.[3]

16.1.1 *Repeatability Limit (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20.

16.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

16.1.1.2 Repeatability limits are listed in Table 1.

16.1.2 *Reproducibility Limit (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20.

16.1.2.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method

16.1.2.2 Reproducibility limits are listed in Table 1.

16.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

16.1.4 Any judgment in accordance with statements 16.1.1 and 16.1.2 would have an approximate 95 % probability of being correct.

17. Keywords

17.1 density; metal powders; outgassing; pycnometry; refractory metal powders; skeletal density; skeletal volume

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:B09-1027. Contact ASTM Customer Service at service@astm.org.