



A Honeywell Company

# Surface Area, Pore Volume, Average Pore Diameter, and Pore Size Distribution of Porous Materials by Nitrogen Adsorption

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## UOP Method 964-11

### Scope

This method is for determining the surface area (SA), total pore volume (PV), average pore diameter (PD), and pore size distribution from 2 to approximately 60 nm (20 to 600 angstroms) of porous materials. The method describes the standard procedure for analysis, data collection and reporting using a commercially available instrument.

### References

- E. Barrett, L. Joyner and P. Halenda, *J. Amer. Chem. Soc.*, **73**, 373 (1951)
- S. Brunauer, P. Emmett, and E. Teller, *J. Amer. Chem. Soc.*, **60**, 309 (1938)
- I. Langmuir, *J. Amer. Chem. Soc.*, **40**, 1361 (1918)
- UOP Method 999, "Precision Statements in UOP Methods," [www.astm.org](http://www.astm.org)

### Outline of Method

The sample is degassed using heat and vacuum. After weighing, it is cooled in liquid nitrogen. The amount of nitrogen gas adsorbed on the sample is measured at various predetermined pressures. The instrument computer processes the data and calculates surface area, total pore volume, average pore diameter, and pore size distribution based on the isotherm. The results are printed and/or plotted as desired (see *Report*).

### Definitions

*BET surface area*, estimated surface area for non-porous materials using the equation derived by Brunauer, Emmett, and Teller, surface area calculated at  $P/P_0 = 0.10, 0.15, 0.20, 0.25, 0.30$

- BET surface area* can be calculated at  $P/P_0$  points other than the standard above.  $P/P_0 = 0.03$  and  $0.3$  are common for certain materials (See *Appendix B*).

*Langmuir surface area*, estimated surface area for microporous materials using the equation derived by Langmuir, surface area calculated at  $P/P_0 = 0.10, 0.15, 0.20, 0.25, 0.30$

<p>IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).</p>
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$P$ , endpoint pressure for a specific data point

$P_0$ , vapor pressure of the adsorbate gas

$P/P_0$ , relative pressure of the adsorbate

$t$ -Plot, plot of statistical thickness of the adsorbed layer of gas versus the adsorbed volume of the adsorbate gas

## Apparatus

References to suppliers and catalog numbers are included as a convenience to the method user. Other suppliers may be used.

*Balance*, analytical, readable to 0.0001 g

*Crucible*, high form, 5-mL, porcelain, Fisher Scientific, Cat. No. 07-965B, with cover, Fisher Scientific, Cat. No. 07-970C, optional, see *Procedure, Sample Pretreatment*, Step 3, bullet

*Desiccator*, 160-mm ID, and porcelain plate, Fisher Scientific, Cat. Nos. 08-632 and 08-641A, respectively, optional, see *Procedure, Sample Pretreatment*, Step 3, bullet

*Dewar*, liquid nitrogen

*Gloves*, heat resistant, Zetex™, VWR, Cat. No. 32885-407

*Gloves*, cryogenic, VWR, Cat. No. 32885-757 (for size large)

*Muffle furnace*, capable of at least 500°C

*Regulator*, helium, two-stage, high purity, delivery pressure range 15 -200 kPa (2-30 psi), Matheson Tri-Gas, Model 3121-580

*Regulator*, nitrogen, two-stage, high purity, delivery pressure range 15 -200 kPa (2-30 psi), Matheson Tri-Gas, Model 3121-580

*Sorption analyzer*, automatic, any commercially available computer controlled system is adequate, with the following accessories. Micromeritics and Quantachrome are two suppliers of this equipment.

*Caps or stoppers*, sized to fit sample cells (will differ depending on manufacturer)

*Sample cells*, pellet and powder

*Stem inserts*, void volume reducers (optional)

*Timer*, 10 minute to 2 hour

*Tongs*, stainless steel, crucible, Fisher Scientific, Cat. No. 15-186, optional, see *Procedure, Sample Pretreatment*, Step 3, bullet

*Vacuum oven*, laboratory, capable of 150°C, with pump

## Reagents and Materials

References to suppliers and catalog numbers are included as a convenience to the method user. Other suppliers may be used.

*Glass wool*, VWR, Cat. No. EM-GX0090-2. Glass wool is not needed for most samples, but may be used to retain powder samples when vacuum is applied.