



A Honeywell Company

Density of Powders and Solids by Helium Displacement

UOP Method 851-08

Scope

This method is for determining true density of powders and solids by displacement of helium gas. This method is written for use with a Micromeritics AccuPyc 1330 Pycnometer; however, equivalent equipment may be used.

References

Owners Manual and Quick Reference Guide, AccuPyc 1330 Pycnometer, Micromeritics Corporation
UOP Method 999, "Precision Statements in UOP Methods," www.astm.org

Outline of Method

The pycnometer works by measuring the pressure drop of a gas as it is expanded into a known volume. The observed pressure change is used to compute the volume of the sample. The measured sample volume excludes all open pore structure as the gas molecules will rapidly fill the pores of the sample; only the truly solid phase of the sample displaces the gas.

If pretreatment of the sample such as calcination or outgassing is required, it must be carried out prior to analysis.

Apparatus

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

Balance, readability, 0.1-mg

Helium Pycnometer, Micromeritics, AccuPyc 1330

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

Reagents and Materials

Cloth, lint-free

Helium, gas, cylinder, 99.995% minimum purity

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).

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Procedure

The analyst is expected to be familiar with general laboratory procedures, the technique of pycnometer measurement, and the equipment being used.

Set up the Micromeritics AccuPyc 1330 Helium Pycnometer as outlined in the manufacturer's instruction manual. Calibration volumes are included with the instrument. Be certain that gauges are calibrated and properly zeroed before proceeding.

Specific Instructions for Micromeritics AccuPyc 1330 Helium Pycnometer

The following detailed procedure for using the Micromeritics AccuPyc 1330 Pycnometer describes the technique for making measurements and is used for the empty cell as well as the cell containing the sample. If a different instrument is used, follow instructions supplied with that instrument.

1. Ensure that the instrument is turned on and that the helium is flowing to it.
2. Remove the cell chamber cap.
 - Do not remove the cell chamber cap when the pycnometer is pressurized.
3. Remove the sample cup from the chamber and obtain a tare weight.
4. Fill sample cup approximately $\frac{3}{4}$ full with sample and reweigh. See *Note 1*.
5. Subtract the tare weight from the filled sample cup and record the sample weight to the nearest 0.1 mg.
6. Insert the sample cup into the cell chamber.
7. Replace the cell chamber cap.
8. Press ANALYZE.
 - For the specified instrument, ANALYZE is selected by pressing the alternate function (blank white) key and then the 4 key. The white function key correlates to white lettering above the numbered keys.
9. Key in the sample identification, numbers only.
10. Press ENTER.
11. Key in the sample weight.
12. Press ENTER.
13. Press ENTER again when CHAMBER INSERT appears.
14. Press ENTER one more time to start the analysis.
 - The instrument is set up to purge the sample and chamber 20 times before the analysis starts.
 - Once the analysis starts it will take up to 99 runs if needed, and will stop automatically once it has 5 consecutive readings without change.
 - The report prints at the end of the run.
15. Remove the cell chamber cap. The instrument will beep once it has depressurized itself and is safe to open.
16. Remove the sample cup from the chamber and clean the sample cup.
17. For further analyses, repeat procedure starting with Step 3, above. When the analyses are finished, replace the clean sample cup in the sample chamber for safe keeping.

Calculations

All calculations are performed by the instrument using the sample weight as entered.

Report results to the nearest 0.001 g/cm³.

Notes

1. If there is insufficient sample to fill the cup $\frac{3}{4}$ full, about 8 mL, an aluminum sleeve with a smaller inside diameter can be used to reduce the available volume. With this adaptation, as little as 2 mL of sample can be used for analysis. The instrument must be recalibrated with new sleeve in place, and some loss of precision would be expected.
2. Before setting regulator pressure, make sure the tank pressure for the gas regulator is at least 200 psig. Pressures less than 200 psig may cause the sample to be inadequately saturated, resulting in inaccurate data or termination of analysis.
3. The cell chamber and cap must be kept clean at all times. Particles on the cap seating surface, in the sample cup, under the sample, or clinging to the sample chamber wall may cause inaccurate results. Inspect the cell and cap before each use. Use a lint-free cloth to remove any dust or particles.
4. Keep the cap on the cell chamber except when actually inserting or removing a sample. If the chamber remains uncapped, temperature instability will occur which could affect analysis results.

Precision

Precision statements were determined using UOP Method 999.

Repeatability and Site Precision

A nested design was carried out for determining the density of three samples with two analysts in one laboratory. Each analyst carried out tests on two separate days, performing two tests on each sample each day (three tests on each day for Sample 3). The total number of tests was 36. The precision data are summarized in Table 1. Two tests performed by the same analyst on the same day should not differ by more than the repeatability allowable difference with 95% confidence. Two tests performed in one laboratory by different analysts on different days should not differ by more than the site precision allowable difference with 95% confidence.

Table 1
Repeatability and Site Precision, g/mL

<u>Sample</u>	<u>Mean</u>	<u>Repeatability</u>		<u>Site Precision</u>	
		<u>Within-Day esd</u>	<u>Allowable Difference</u>	<u>Within-Lab esd</u>	<u>Allowable Difference</u>
1	1.34	0.0002	0.001	0.0006	0.004
2	1.39	0.0001	0.001	0.0002	0.001
3	1.96	0.0050	0.016	0.0056	0.018
4	3.29	0.0028	0.011	0.0039	0.015

Samples 1 and 2 are non-porous materials (plastic chips). Sample 3 is a zeolite. Sample 4 is a catalyst support. Precision is typically better on non-porous materials than on porous materials.

The data in Table 1 are a short-term estimate of repeatability. When the test is run routinely, a control standard and chart should be used to develop a better estimate of the long-term repeatability.

Reproducibility

There is insufficient data to calculate reproducibility of the test at this time.

Time for Analysis

The elapsed time and labor requirement for a single analysis are identical, 0.8 hour.

Suggested Suppliers

Matheson Tri-Gas, 166 Keystone Drive, Montgomeryville, PA 18936 (215-648-4000)
www.matheson-trigas.com

Micromeritics Instrument Corporation, One Micromeritics Drive, Norcross, GA 30093-1877
(770-662-3620) www.micromeritics.com