

DENSITY OF POWDERS AND SOLIDS BY HELIUM DISPLACEMENT

UOP Method 851-84

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 Quantachrome Null-Pycnometer, however, equivalent equipment is satisfactory. If pretreatment of the sample such as calcination or outgassing, is required, it must be carried out prior to analysis.

OUTLINE OF METHOD

A volume of gas is added to the sample cell of a Null-Pycnometer and the resultant pressure is measured when a differential gauge is nulled. This procedure is repeated with the same sample cell containing a weighed sample. From the two readings the sample volume, and thereby true density, can be calculated. The results are reported in g/cm^3 .

APPARATUS

Balance, readability, 0.1-mg

Null-Pycnometer, Quantachrome Corp., Model PY-5, or equivalent

Regulator, helium, Matheson, Model 8, GCA580, or equivalent

Valve, adjustable safety relief, 69-207 kPa, brass, Air Products, Cat. No. E33-41121, or equivalent

REAGENTS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available, unless otherwise specified.

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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UOP Methods are available through ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken PA 19428-2959, United States. The Methods may be obtained through the ASTM website, www.astm.org, or by contacting Customer Service at service@astm.org, 610.832.9555 FAX, or 610.832.9585 PHONE.

PROCEDURE

Set up the Null-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.

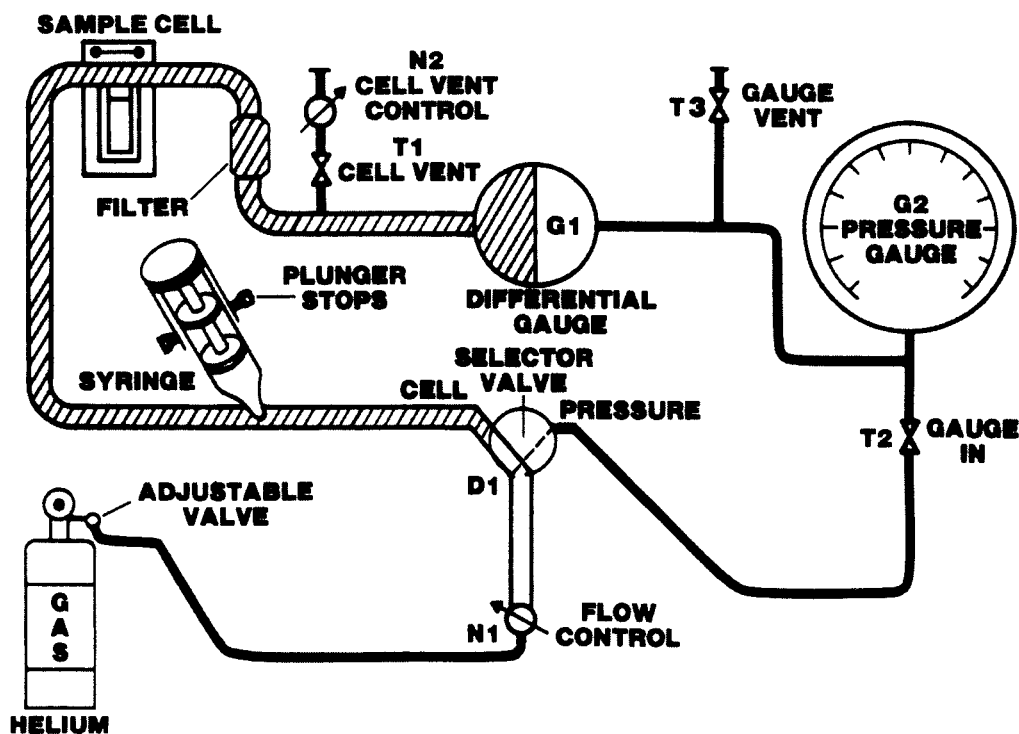
With the cell empty, follow the manufacturer's recommended procedure to obtain a reading on the pressure gauge. This is recorded as P_2 . Repeat this procedure until three readings are within ± 0.01 psig (0.069 kPa). Open the cell holder, remove the sample cell and weigh it to the nearest 0.1 mg. Place a 75-100 cm^3 sample (see Note 1) in the cell and reweigh. Record the mass of the sample. Follow the same procedure, this time with the sample in the cell. Record the reading as P_2' . Repeat the procedure until three readings are within ± 0.01 psig (0.069 kPa).

Specific Instructions for Quantachrome Null-Pycnometer

The following detailed procedure using the Quantachrome Null-Pycnometer, see Figure, describes the technique for making measurements and is used for the empty cell as well as the cell containing the sample.

Set the Adjustable Valve on the helium inlet line to 20 ± 3 psig (138 kPa). Turn the Selector Valve (D_1) to "Pressure". Open Cell Vent (T_1) and Cell Vent Control (N_2). Close Gauge In (T_2) and open Gauge Vent (T_3). Close Flow Control (N_1), (see Note 2). Both sides of the differential gauge are open to ambient atmosphere.

Turn the Selector Valve (D_1) to "Cell". Open the Flow Control valve (N_1) slowly, permitting the pointer on the Differential Gauge (G_1) to deflect up scale to approximately 40 scale divisions (10 kPa). Slowly lift the Syringe plunger to fill the syringe. Wait 15 ± 2 seconds and turn Selector Valve (D_1) to "Pressure". Close Flow Control (N_1). Wait 20 ± 2 seconds after the Differential Gauge (G_1) returns to zero. The sample system and syringe are now filled with helium at ambient pressure.



Schematic Diagram of Quantachrome Instrument

Close Cell Vent (T₁) and depress the handle against the cabinet for 5 seconds to relieve the pressure built up from the valve stem action. Close Cell Vent Control (N₂). The Pressure Gauge (G₂) should read zero; if not, refer to manual on zeroing gauges. The sample system is closed.

Depress the Syringe plunger completely and lock the plunger by gently tightening the knob. Close Gauge Vent (T₃). A reproducible volume of helium has been introduced into the closed sample cell system.

Partially open Gauge In (T₂) by depressing the handle against the cabinet. While T₂ is depressed, adjust Flow Control (N₁) to control the rate of pressure increase on Pressure Gauge (G₂). Differential Gauge (G₁) will show a decrease. As the pointer on the Differential Gauge (G₁) approaches zero, slowly close Flow Control (N₁). When the pointer on the Differential Gauge (G₁) crosses the zero mark, release Gauge in (T₂) to close. The Differential Gauge (G₁) has been nulled.

Read Pressure Gauge (G₂) to the nearest 0.01 psig (0.069 kPa) and record the value as P₂ (value for empty cell) or P₂' (value with sample in cell). This is the pressure exerted when the helium in the syringe is compressed into the system.

Open Gauge Vent (T₃). Close the Cell Vent Control (N₂). Open Cell Vent (T₁) and slowly open Cell Vent Control (N₂) to allow pressure on the Differential Gauge (G₁) to gradually approach zero. The instrument is ready for the next measurement starting with "Turn Selector Valve (D₁) to 'Cell'."

CALCULATIONS

Calculate the volume of the sample taken for analysis as follows:

$$V_s = V_c \left(1 - \frac{P_2}{P_2'} \right)$$

where:

- P₂ = pressure of empty cell, psi (kPa)
(reading on Pressure Gauge [G₂])
- P₂' = pressure of cell with sample, psi (kPa)
(reading on Pressure Gauge [G₂])
- V_c = volume of empty cell, cm³
(supplied by manufacturer)
- V_s = volume of sample, cm³

Calculate the true density as follows:

$$\text{Density, g/cm}^3 = \frac{W}{V_s}$$

where:

- V_s = as previously defined
- W = mass of sample, g

Report results to the nearest 0.01 g/cm³

NOTES

1. If insufficient sample is available for analysis, an aluminum sleeve can be inserted in the cell to reduce its volume. Both P_2 and P_2' must be determined with the sleeve in place. In this fashion, as little as 5 cm³ of sample can be used for analysis.

2. Valves, N_1 and N_2 are needle valves. Valves T_1 , T_2 and T_3 are toggle valves which have three operating positions; fully open, fully closed and partially open when the handle is pressed down against the cabinet.

PRECISION

Based on 6 replicate determinations, the estimated standard deviations (esd) were calculated and are shown in the following table. Results by the same operator should not differ by more than the allowable difference shown (95% probability).

<u>Sample Volume, mL</u>	<u>Density Determined, g/cm³</u>	<u>esd, g/cm³</u>	<u>Allowable Difference, g/cm³</u>
5 - 12	2.53	0.029	0.11
75 - 100	2.38	0.007	0.03

TIME FOR ANALYSIS

The elapsed time and labor requirement for a single analysis are identical, 0.75 hour.

REFERENCE

“Theory and Operating Instructions”, Null-Pycnometer, Model PY-5, Quantachrome Corporation

SUGGESTED SUPPLIERS

Air Products and Chemicals, Inc., Specialty Gas Dept., Box 538, Allentown, PA 18105

Matheson, P.O. Box 96, Joliet, IL 60434

Quantachrome Corporation, 6 Aerial Way, Syosset, NY 11791