



Density of Viscous Liquids and Semi-Solid Hydrocarbons by Nitrogen Displacement

UOP Method 1025-18

Scope

This method is for determining the true density of viscous liquids and semisolid hydrocarbons by displacement of nitrogen gas. This method is written for use with a Micromeritics AccuPyc II 1340 Pycnometer; however, equivalent equipment may be used.

References

- Owner's Manual and Quick Reference Guide, AccuPyc II 1340 Pycnometer, Micromeritics Corporation
- UOP Method 851, "Density of Powders and Solids by Helium Displacement," www.astm.org
- 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.

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

Gas Pycnometer, Micromeritics, AccuPyc II 1340

Oven, capable of operation up to 150°C

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

Tongs or Tweezers, suitable for removing sample cups from the oven

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. SAFETY DATA SHEETS (SDS) OR EXPERIMENTAL SAFETY DATA SHEETS (ESDS) FOR ALL THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).
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Reagents and Materials

Cloth, lint-free

Nitrogen, gas, cylinder, 99.995% minimum purity, local supply

Sample Cup, Aluminum, 3.3 cc, Micromeritics Cat no. 134-25600-00

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 II 1340 Gas 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 II 1340 Nitrogen Pycnometer

The following detailed procedure for using the Micromeritics AccuPyc II 1340 Pycnometer describes the technique for making measurements and is used for the empty cell as well as the cell containing the sample within a disposable cup. If a different instrument is used, follow instructions supplied with that instrument.

1. Ensure that the instrument is turned on and that the nitrogen is flowing to it.
2. Ensure that the cell cooling is turned on and temperature is stable. Depending on instrument configuration, this could be from a circulating water bath or direct thermoelectric cooling.
3. Place samples in an oven to melt, if solid at room temperature. Sample should be liquid and pourable after heating. Stir thoroughly to mix before taking a portion of the sample.
4. Obtain a tare weight from an empty, disposable sample cup.
5. Fill sample cup approximately $\frac{3}{4}$ full with sample and reweigh.
6. Subtract the tare weight from the filled sample cup and record the sample weight to the nearest 0.1 mg.
7. If the sample was solid at room temperature, then place the cup back into the oven and re-melt for 15-20 minutes to allow any bubbles or voids to fill.
8. Cool the sample to room temperature before placing it into the measurement cell.
9. Remove the cell chamber cap.
 - Do not remove the cell chamber cap when the pycnometer is pressurized.
10. Insert the sample cup into the cell chamber.
11. Replace the cell chamber cap.
12. On the PC, select the instrument unit and click sample analysis. Select the correct file in the directory column or create a new file.
13. Enter necessary sample information and correct sample mass.
 - Ensure the 3.5 cc chamber insert option is selected.
14. Click start to begin analysis.
15. Obtain data from the report printed on the screen.

16. The instrument will beep once it has depressurized itself and is safe to open. Remove the cell chamber cap.
17. Remove the sample cup from the chamber and dispose of appropriately.
18. For further analyses, repeat procedure starting with Step 3, above.

Calculations

All calculations are performed by the instrument using the sample weight as entered. Report results to the nearest 0.001 g/cm³.

$$V_{\text{samp}} = V_{\text{cel}} - \frac{V_{\text{exp}}}{\frac{P_1}{P_2} - 1}$$

$$D_{\text{samp}} = \frac{M_{\text{samp}}}{V_{\text{samp}}}$$

where

- V_{cel} = sample chamber volume
- V_{exp} = expansion chamber volume
- V_{samp} = sample volume
- M_{samp} = sample mass
- D_{samp} = sample density
- P_1 = gauge pressure after fill
- P_2 = gauge pressure after expansion

Notes

1. 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.
2. The cell chamber and cap must be kept clean and lightly greased 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. Very lightly grease O-ring on cell chamber cap.
3. Keep the cap on the cell chamber except when 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 one sample with two analysts in one laboratory. Each analyst carried out tests on two separate days, performing two tests on each sample each day. 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>
A	1.0037	0.00008	0.0003	0.00034	0.0021

The data in Table 1 is 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, 1 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