QUANTACHROME

PYCNOMETERS

Automatic

True Density Analysis of Powders, Foams and Bulk Solids

PYCNOMETRY

"Pycnometry" is derived from the Greek word pyknos, which has long been identified with volume measurements. The pycnometers from Quantachrome are specifically designed to measure the true volume of solid materials by employing Archimedes' principle of fluid (gas) displacement and the technique of gas expansion.

Ideally, a gas is used as the displacing fluid since it penetrates the finest pores assuring maximum accuracy. For this reason helium is recommended, since its small atomic dimension enables entry into crevices and pores approaching two Angstrom ($2x10^{-10}$m). Its behavior as an ideal gas is also desirable. Other gases such as nitrogen can be used, often with no measurable difference.

Applications

Quantachrome pycnometers are used for research, development and quality control in such diverse industries as carbon black, catalysts, cement, ceramics, charcoals, cosmetics, desiccants, fertilizers, fibers, fillers, insulating and structural foams, powdered foods, ion exchange resins, minerals such as alumina, silica, titania and others, nuclear fuels, petrochemicals, pharmaceuticals and powdered metals. Pycnometry can even determine the percentage of solids in a slurry.

The **PENTAPYCNOMETER** and the **ULTRAPYCNOMETER 1000** from Quantachrome are the ultimate instruments for measuring the true volume and density of powders, foams and bulk solids. A wide range of sample cell sizes are easily interchanged to accommodate many different samples. Calibration, sample conditioning, operation of valves and calculation of results are completely automatic. Samples are quickly and automatically analyzed as many times as necessary to achieve the user desired % deviation from mean at the specified number of runs. Should the deviation setting be too narrow, the analysis terminates upon reaching an operator specified maximum number of runs. The results are printed automatically, freeing the operator to perform other laboratory tasks.

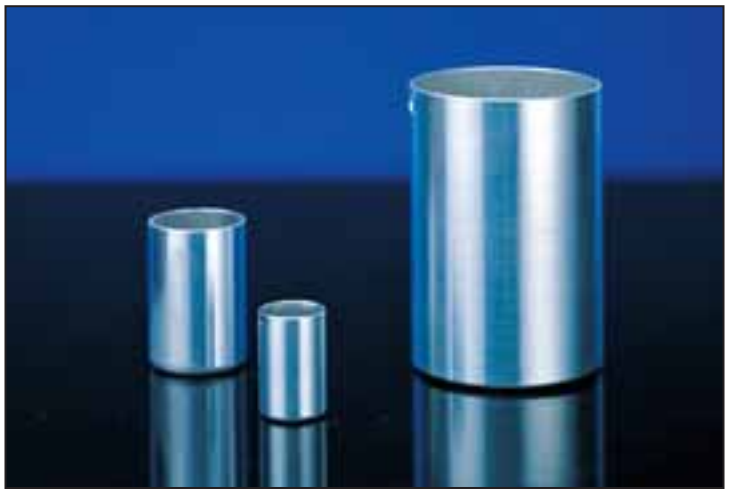
The **PENTAPYCNOMETER** permits up to five samples to be loaded and then purged simultaneously. Each of the five samples is then automatically analyzed in sequence with no operator involvement. Individually adjustable flow rates for each cell provide maximum flexibility of operation.

For laboratories with lower analytical throughput needs, the single sample station **ULTRAPYCNOMETER 1000** provides the same ease of operation and high performance as the **PENTAPYCNOMETER**.

- For measurements that require a fixed, known temperature, a temperature control option is available for both the **ULTRAPYCNOMETER 1000** and the **PENTAPYCNOMETER** (requires external bath/circulator, sold separately).
- For measurements in a hazardous environment, such as a nuclear "hot cell", a **remote operation ULTRAPYCNOMETER 1000** is available.
- For measurement of foams, a specialized version called the **UltraFoam™** pycnometer is available. It is a system for automatic measurement of open and closed cell content with automatic functions for the analysis of cell compressibility and cell fracture according to ASTM D-6226.
- For measurements of extremely small amounts of material, the **Micro ULTRAPYCNOMETER** provides three interchangeable cell sizes of 4.5 cm^3, 1.8 cm^3 and 0.25 cm^3.

Sample Cells	Nominal Volume	Internal Diameter	Internal Length
Standard sample cells	135 cm^3	49 mm	75 mm
	50 cm^3	40 mm	39 mm
	10 cm^3	24 mm	24 mm
Optional Microcell	4.5 cm^3	16 mm	25 mm
Optional Mesocell	1.8 cm^3	13 mm	13 mm
Optional Nanocell	0.25 cm^3	8 mm	6.5 mm

The sample cells listed above are aluminum.
All cells are available in stainless steel for durability.
Non-elutriating cells are available for fine powders.



Gas Input and Vacuum Fittings

Foods and Pharmaceuticals

Catalysts and Ceramics

ERATION SERIES

CURACY REPRODUCIBILITY

Feature Benefits

Automatic Functions - Operating simplicity is achieved by user prompts or instructions which are automatically presented on the alphanumeric display. Measurements and printout of results are totally automatic. Continuous self-diagnostics monitor and signal fault conditions that may arise. The transducer is reset to zero prior to each run. Front panel LEDs display the operational status at all times.

Sample and User ID - In the analysis parameters, alphanumerics may be added to uniquely identify both the sample and operator.

Temperature - Sample temperature is displayed and printed to ±0.1 °C. This feature is important for: (a) verifying operation at the calibration point or, (b) making corrections when analyzing larger quantities of materials whose density varies significantly with temperature. Optional temperature controlled capability available (requires external bath/circulator–sold separately).

Target Pressure - The measurement of pressure-deformable cellular foams (insulation) is made possible by this feature. A user can conveniently reduce the target pressure typically from around 18 psi to as low as 0.5 psi. A special version, the **UltraFoam**™ pycnometer, automatically scans for the optimum pressure and performs calculations of open and closed cell content with or without ASTM correction for cut cells.

Sample Purge - Before analysis, samples are automatically conditioned to remove contaminants and trapped air. The user has a choice to purge by a *continuous flow* to prevent elutriation (blowing out) of fine particles, by a pulse mode more suitable for coarse powders or bulk solids. The **PENTAPYCNOMETE**R purges all five stations simultaneously, thus requiring no more time than for a single sample.
The **ULTRAPYCNOMETE**R has the added capability to purge the sample by vacuum for a "user selectable" time, (vacuum pump supplied separately).

Repeat Run Mode - This feature eliminates the need to reenter the same set of analysis parameters prior to each run. It allows one to quickly change sample weight and sample ID, or simply to rerun a sample by a double keystroke.

Useful Statistics - For three or more averaged measurements the % coefficient of variance and the standard deviation of the volume and the density are printed out. This allows a relative comparison of samples from run-to-run and a precise assessment of the absolute variation of the sample being measured.

NIST Traceability - Pycnometer volume calibration spheres can be provided with a formal Report of Calibration from the National Institute of Standards and Technology.

Computer Software - To capture printed output, automatic pycnometers are available with Windows® based PC software that provides disk storage (ASCII format) of data along with all user entered information. Archived files can be recalled for review, printing and importation to other programs.

Balance Port - Permits interface with configurable analytical balance for automatic transfer of sample weight. Eliminates risk of transcription error.

Powder Metals and Construction

Foams and Fibers

NIST traceable calibration spheres.

UltraFoam™

Automatic Gas Pycnometer for the Characterization of Foams

A complete sytem for the automatic measurement of:

- **Open Cell Content**
- **Closed Cell Content**
- **ASTM Method D6226**
  (with or without correction for cut cells)

With special pressure related functions for the automatic analysis of:

- **Cell Compressibility**
- **Cell Fracture**

Featuring interchangeable sample cells, automatic reporting and PC archiving of data.

As seen at the SPE's Foams 2004 Conference

The cell content of porous materials correlates with performance attributes such as strength, fluid exclusion (or acceptance) and insulating properties. Closed cells impart water resitance, thermal insulation, buoyancy and resilience. Open cells determine properties related to filtration, acoustics and wicking.

To such ends, the analysis of cell content in porous materials is best performed by gas displacement...gas pycnometry. The gas, usually nitrogen or helium, quickly conditions the sample by carrying away blowing agents, moisture, air, etc. as it probes all cell cavities connected to the surface. Single or multiple measurements can be automatically performed, each taking but one or two minutes.

The standard technique used by the UltraFoam™ pycnometer is that of gas expansion from a calibrated sample chamber into a reference volume. The solid and closed cell volume of the sample is calculated from the relationship of the calibrated cell volumes and the pressures before and after expansion. The closed cell percentage is calculated from the solid volume and the measured geometric volume of a rectangular or cylindrical sample. Open cell % is calculated by difference (open cell % = 100% - closed cell %). Density is calculated from mass (weight)/volume and is reported as grams/cm^3.

ADVANCED METHODS FOR DIFFERENT APPLICATIONS:

Correction For Cut Cells

In ASTM D6226, a corrected percentage of open cells is estimated by remeasuring the volume of a cube *after sectioning into eight parts*. The UltraFoam pycnometer programming provides a convenient way to automate this procedure. The printed report then gives both the Open Cell % and the Corrected Open Cell % values.

Cell Compressibility

The UltraFoam pycnometer offers a convenient feature that extends its range of usefulness for less rigid foams. By automatically increasing pressure stepwise, the resulting series of Compression % data provides a rational basis to determine:
1. Compression profile/ characteristics for quality control,
2. If there is an optimum pressure for the most accurate Open Cell % and Closed Cell% readings.

Cell Fracture

In a similar manner, the UltraFoam pycnometer has the important analytical ability for rigid foams that have fragile cell walls. In this mode, each step of increasing pressure is tested to see if the pressure change caused a *permanent* decrease in Closed Cell %. The data series of Fracture % versus pressure thus presented provides a rational basis to determine:
1. Cell fracture profile/ characteristics for quality control,
2. If there is an optimum pressure for best accuracy.

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