

# Gas Pycnometers

True Density Analysis of Powders, Foams and Bulk Solids



Gas Pycnometers



Catalysts



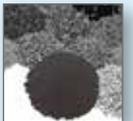
Ceramics



Energy



Carbon



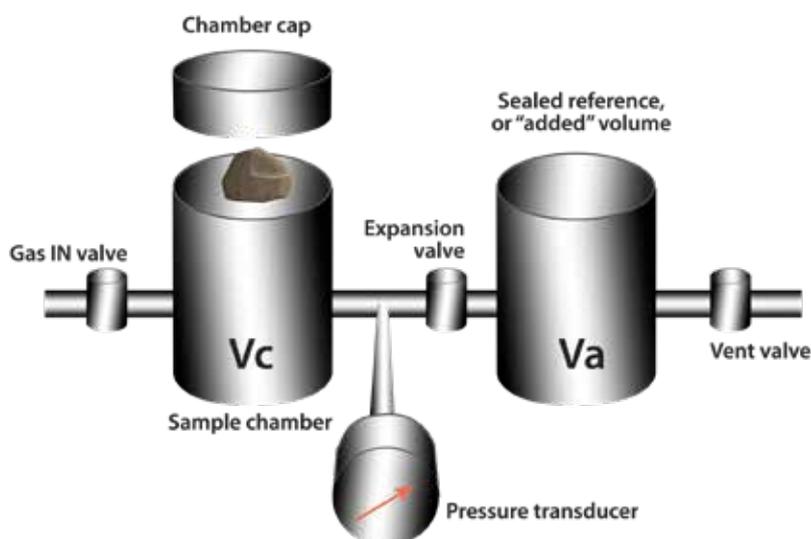
Pharma



## Gas Pycnometry

### Introduction

The word "Pycnometry", derived from the Greek word *pyknós*, meaning 'dense', has long been identified with volume measurements. Quantachrome's automatic gas pycnometers have long been identified as the instruments of choice to accurately measure the true density of solid materials by employing Archimedes' principle of fluid displacement, and Boyle's Law of gas expansion. An inert gas, rather than a liquid, is used since it will penetrate even the finest pores and eliminate the influence of surface chemistry. This ensures quick results of the highest accuracy. Helium is generally used as the displacement gas due to its size and generally inert behavior. Other gases, such as nitrogen, are also routinely used with no measurable sacrifice of performance.



### Principle of Operation

A sealed sample chamber of known volume is pressurized to a target pressure with the displacement gas. Once stabilized, this pressure is recorded. A valve is then opened allowing the gas to expand into a reference chamber whose volume is also known. Once stabilized, this second pressure is recorded. This pressure drop ratio is then compared to the behavior of the system when a known volume standard underwent the same process. Because the measurement depends only upon the pressure drop ratios there is no need for calibration of the pressure transducers.

To ensure accurate results on a gas pycnometer there exists a proper ratio between the volumes of the reference chamber and the volume of the sample chamber. If this ratio is too large or too small, the subsequent pressure-drops created during the analysis will lead to inaccuracies.

This is why most gas pycnometers manufactured by Quantachrome Instruments possess multiple built-in reference volumes. The combination of adjustable sample chamber sizes and multiple internal reference chambers ensures that a single gas pycnometer from Quantachrome Instruments will provide accurate results over a range of volume applications that would require up to three distinct instrument models from competitive manufacturers.

### Benefits of Gas Pycnometry

- Non-destructive analysis
- Fast and accurate results (in as little as 1 minute)
- Reliable and reproducible results
- Wide range of sample volumes and configurations
- Instrument has small footprint
- Uses small amount of gas pressurized to < 20 psig

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## Applications

### Pharmaceuticals

The true density of active and excipient materials is used to determine their composition for both development and process control efforts. See **USP [699]**.

### Powder Coatings

The crystallinity of plastics used in coatings as well as the true density of dry pigment materials are monitored via gas pycnometry measurements. See **ASTM D5965**.

### Cement

The density of cement is used for accurate calculation of powder characteristics and, measured after set up, is important for formulation and stability determination of slurries.

### Petroleum Coke

The true density of calcined petroleum coke is an important quality specification for these materials. See **ASTM D2638**.

### Glass Microspheres

Broken spheres are undesirable. The ratio of intact to fractured spheres is routinely evaluated using gas pycnometry.

### Soils

True density values assist in assessing whether or not certain impurities are present in the composition of soils. See **ASTM D5550**.

### Ceramics and Catalysts

Density values are used in the development, manufacturing, and troubleshooting of refractory materials to confirm desired crystal phase is present and closed porosity is absent. See **ASTM C604**.

### Polymers and Foams

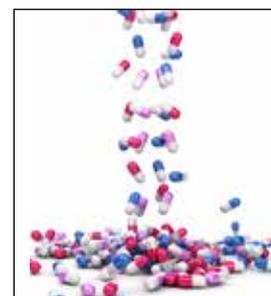
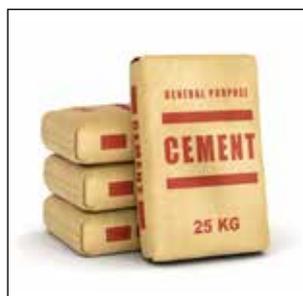
Gas pycnometry is widely used to characterize the relative amounts of crystalline and amorphous phases as well as open and closed cells present in polymer materials. See **ASTM D6226**.

### Dried Film Coatings

The density of dried film coatings is used in the assessment of their volatile organic content. See **ASTM D6093**.

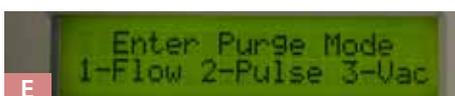
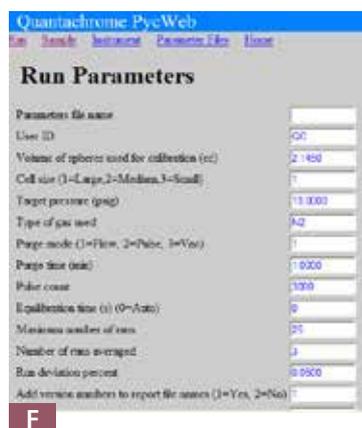
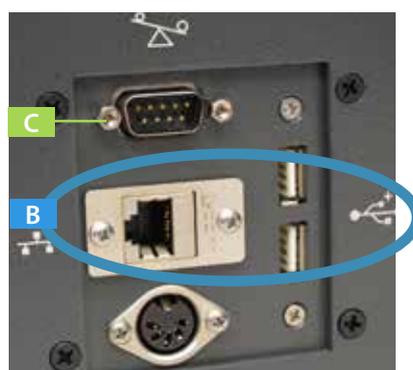
### Metallurgy

The true density of complex metal shapes formed by powder metallurgy is used to track physical properties throughout the processing of such structures. See **MPIF Standard 63**.



## Automatic Gas Pycnometers

The **UltraPyc 1200e** and the **PentaPyc 5200e** from Quantachrome Instruments are the ultimate true volume and true density measurement units due to their reliability, accuracy, reproducibility, and minimal operator involvement. Their wide range of interchangeable sample cell sizes (from 1 to 135 cc) served by a range of appropriate reference volumes make them perfect for either quality control or research and development environments.



### **A** Compact

The **UltraPyc 1200e** is just 31 cm wide, and the **PentaPyc 5200e** can analyze up to five samples in just 42 cm of bench space.

### **B** Access to Results

Detailed reports may be **printed, emailed, or saved to a USB flash drive** immediately following an analysis. Results are retained internally in text and pdf format for access via PC for remote printing, sharing, and archiving.

### **C** Direct Input of Sample Mass

An RS232 port for connection to an electronic balance to obtain the mass directly is included. Mass may also be entered at the keypad or via the PC interface.

### **D** Direct Operation

Analyses can be setup and started using the built-in keypad or PC via ethernet. Menu selections and prompts are presented on a backlit display located on the front of the unit.

### **E** Automatic Sample Preparation

The sample chamber is automatically purged with analysis gas per operator protocol. Vacuum assisted sample preparation is also available on the **UltraPyc 1200e**.

### **F** Customizable Analysis Parameters

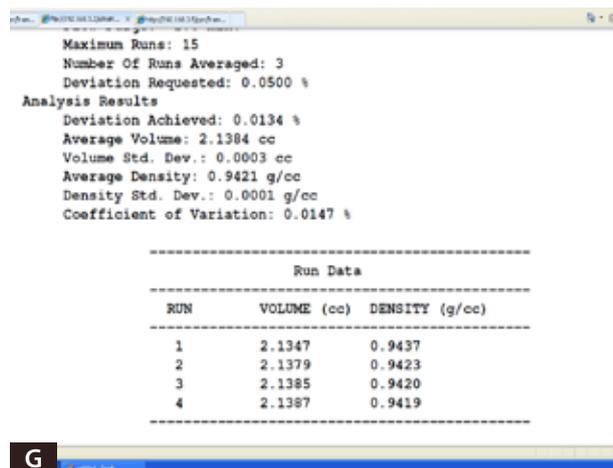
Define sample preparation protocol, number of expansions, repeatability acceptance criteria, equilibrium conditions, and target pressure.

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## Automatic Gas Pycnometers



G



H

### G Automatic Repeated Analysis

Measurements are automatically repeated until a specified number of contiguous measured values agree within a specified deviation. Data averaging and statistical calculations are performed automatically.

### H NIST Pedigree

Calibration spheres can be purchased with a formal Report of Calibration provided by the National Institute of Standards and Technology: Not simply traceable to NIST, but actually measured by NIST.

### Easy to Use

Samples are easy to load and unload. Measurement protocols are easily invoked either at the unit or via a web browser with pre-installed software.

### Fast Initialization

A new analysis requires only that correct sample ID and mass be entered. The entire analysis protocol remains in memory for the next analysis.

### Time Saving Operation

Automatic analysis completely frees the operator for other tasks while the unit works totally unattended. The PentaPyc 5200e can analyze up to five samples.

### Emphasis on Precision and Accuracy

The sample chamber is vented and the pressure transducer is zeroed before every expansion, not just when a fresh sample is initialized. The appropriate expansion chamber is automatically employed.

### Focus on Reproducibility

The pycnometers retain volume calibrations in memory even when powered down – regular adjustment is not required. The system temperature is displayed in reports for verification of suitable and stable measurement conditions.

### Adaptability

Optimal performance is assured by multiple sample cup sizes and multiple expansion chambers for the micro-processor, not the operator, to choose from.

### Pre-Calibrated, Re-Calibrate Any Time

All pycnometers are factory calibrated and ready to use right out of the box. Re-calibration is easy to do with the supplied set of reference volume spheres.

### Automatic Troubleshooting Alerts

Loss of gas pressure, unstable pressure, and other fault conditions are automatically reported to the operator for remedial troubleshooting action.

# Gas Pycnometers

True Density Analysis of Powders, Foams and Bulk Solids



The **UltraPyc 1200e** Automatic Gas Pycnometer is used for the measurement of true density and volume of solids. The **UltraPyc 1200e** features automatic “repeat run” measurement mode until the results fall within a user-specified tolerance or achieve a user-specified maximum number of runs. The appropriate expansion (reference) volume is automatically chosen by a microprocessor according to the sample cell selected. Reports include density and volume results with statistics. In addition, the instrument features a USB printer port, a second USB port for a flash/thumb drive, ethernet port for PC (automatic operation via web browser) or network, RS232 balance interface, and a storage compartment.

Supplied complete with three interchangeable sample cells (10, 50 & 135 cc) and a full set of calibration spheres.

**Dimensions (WxDxH):** 31cm x 54 cm x 22.5 cm  
**Weight:** 14 kg

# Gas Pycnometers

True Density Analysis of Powders, Foams and Bulk Solids

## UltraPyc 1200e

### Analysis Specifications

**Sample Volume:** 0.1 - 135 cm<sup>3</sup>

**Resolution:** 0.0001 g/cm<sup>3</sup>

**± Accuracy and Reproducibility:**

- **Large Sample Cell** (135 cm<sup>3</sup>)  
Volume Accuracy-better than +/- 0.02%  
Repeatability -better than +/-0.01%
- **Medium Sample Cell** (50 cm<sup>3</sup>)  
Volume Accuracy-better than +/- 0.02%  
Repeatability -better than +/- 0.01%
- **Small Sample Cell** (10 cm<sup>3</sup>)  
Volume Accuracy-better than +/- 0.03%  
Repeatability -better than +/- 0.015%

**Analysis Time:** In as little as 1 minute

**Purge Modes:**

- Continuous flow with user selectable time; or
- Pulse with user selectable number of pressurization/depressurization cycles; or
- Vacuum for user selectable time with automatic valve configuration (vacuum pump supplied separately); and
- Flow/Evacuation Rates: User-selectable with precision needle valve.

**Pressure Range:** 1-20 psig

**Gas Requirements:** Compressed gas (preferably ultra high purity helium or nitrogen for helium-permeable samples) regulated to 20 psig (140 kPa).

± Accuracy and reproducibility vary with sample volume and preparation. Values given above are for well-prepared samples with volumes exceeding 50% of the cell volume.





The **PentaPyc 5200e** Automated Gas Pycnometer is utilized for high throughput measurements of true density and volume of solids.

The **PentaPyc** features five analysis ports for simultaneous sample preparation and sequential analysis, and automatic repeat measurement mode until results fall within a user-specified tolerance or achieve a user-specified maximum number of runs.

The appropriate expansion (reference) volume is automatically chosen by a microprocessor according to the sample cell selected. Reports include density and volume results with statistics.

In addition, the instrument features a USB printer port, a second USB port for a flash/thumb drive, Ethernet port for PC (automatic operation via web browser) or network, and RS232 balance interface. Complete with three interchangeable sample cells (10,50 & 135 cc) for each station and full set of calibration spheres.

**Dimensions (WxDxH):** 42 cm x 58 cm x 32 cm

**Weight:** 33 kg

# Gas Pycnometers

True Density Analysis of Powders, Foams and Bulk Solids

## PentaPyc 5200e

### Analysis Specifications

**Sample Volume:** 0.1 - 135 cm<sup>3</sup>

**Resolution:** 0.0001 g/cm<sup>3</sup>

**± Accuracy and Reproducibility:**

- **Large Sample Cell** (135 cm<sup>3</sup>)  
Volume Accuracy-better than +/- 0.02%  
Repeatability -better than +/-0.01%
- **Medium Sample Cell** (50 cm<sup>3</sup>)  
Volume Accuracy-better than +/- 0.03%  
Repeatability -better than +/- 0.015%
- **Small Sample Cell** (10 cm<sup>3</sup>)  
Volume Accuracy-better than +/- 0.2%  
Repeatability -better than +/- 0.1%

**Analysis Time:** In as little as 1 minute

**Purge Modes:**

- Continuous flow with user selectable time; or
- Pulse with user selectable number of pressurization/depressurization cycles; and
- Flow/Evacuation Rates: User-selectable with precision needle valve.

**Pressure Range:** 1-20 psig

**Gas Requirements:** Compressed gas (preferably ultra high purity helium or nitrogen for helium-permeable samples) regulated to 20 psig (140 kPa).

± Accuracy and reproducibility vary with sample volume and preparation. Values given above are for well-prepared samples with volumes exceeding 50% of the cell volume.



## Special Versions

### Foam Measurements

For measurement of foam materials, there are two specialized versions of our automatic gas pycnometers. The **UltraFoam 1200e** and **PentaFoam 5200e** instruments provide automated measurements of Open-Closed Cell content, cell compressibility, and cell fracture. Both units are supplied with a Foam Cutting Kit (see Accessories).



### Micro-UltraPyc 1200e

For the measurement of extremely small amounts of material, the **Micro-UltraPyc 1200e** features smaller expansion chambers and smaller sample cups (0.25, 1.8, 4.5 cm<sup>3</sup>) than the standard **UltraPyc 1200e**. This ensures accurate measurement of these very small sample amounts.



### Temperature Control Option

For measurements that require a fixed, known temperature, All gas pycnometers manufactured by Quantachrome Instruments have the hardware necessary for connection to an external temperature controller bath.

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## Available Accessories

### Gas Regulator

Quantachrome Instruments recommends the use of a 2-stage gas (Helium or Nitrogen) regulator assembly complete with CGA fitting and an isolation valve on the output fitted with a 1/8" connector for attachment to the gas line supplied with its pycnometers. This assembly is available for purchase through Quantachrome Instruments.

### Temperature Control Module

A compact yet powerful thermoelectric heater/chiller circulates liquid to the heat exchange system built into pycnometers equipped with the Temperature Control Option.

### Vacuum Pump

For use with **UltraPyc 1200e** or **Micro-UltraPyc 1200e** models should vacuum degassing be desired. This compact two-stage rotary vane vacuum pump comes complete with hose and fittings necessary for connection to these pycnometers when purchased from Quantachrome Instruments.

### Sample Cups and Adapters

The sample cups provided for use with each pycnometer are fabricated to close tolerance from stainless steel. They are durable and easy to clean. Delrin<sup>®</sup> versions are also available. Various cup sizes are available to best fit your specific application needs.



### Micro-Cell Option

For use with **UltraPyc 1200e** and **PentaPyc 5200e** instruments to extend the measurement range of these instruments, this kit contains 0.25, 1.8, and 4.5 cm<sup>3</sup> sized sample cups and all necessary adapters along with calibration spheres and removal tools in a padded wooden box.



### Non-Elutriating Sample Cells

For very fine easily aerosolized materials, such as carbon black toner or fly ash samples, there are "Non-Elutriating" versions of the sample cups. These cups have perforated lids which sit on top of the sample cup without need for o-rings, grease, clips, or springs.



### Foam Cutting Kit

Includes a sample holder with guide slots for reproducible preparation of 1" cubes of rigid foams as required in ASTM D6226. Also included are an electric knife and a circular punch for obtaining other "regular" shaped samples. This is supplied as standard with **UltraFoam 1200e** and **PentaFoam 5200e** instruments, but can be purchased separately.

### Percent Porosity Kit

For use on a Quantachrome Instruments tapped density analyzer such as the **AutoTap** or **Dual AutoTap** instruments. Includes pycnometric powder and graduated measurement column. Calculations of percent porosity made off line in conjunction with gas pycnometry results.





Renowned innovator for today's porous materials community. The quality of Quantachrome's after sales service support is the reason we are proud to maintain life time relationships with our customers.

## Field Service

Our global service staff assure you that Quantachrome Instruments will continue to be the reliable engines of material characterization laboratories. We offer you the flexibility of choosing from service contracts tailored to provide you with the response time, service package, and spare parts discounts that best fit your needs.

## Spare Parts

Quantachrome spare parts are certified to work with our instruments. We provide rapid response spare parts orders, and keep large inventories of replacement parts and hardware available.

## Application Lab

Our fully equipped, state-of-the-art powder characterization laboratory (email: [application.qt@anton-paar.com](mailto:application.qt@anton-paar.com)), provides the option of contracting for expert testing services. Laboratory services are also available to validate the applicability of our products prior to your purchase using your actual samples.

## Lifetime Application Support

We view the field support of our instruments as an essential component of our business strategy. Our expert scientists are always available to answer questions on applications, or the use of our instruments. We do this as a standard service regardless of whether you have a service contract with us or not.

## Partners in Science

Quantachrome has a scientific research department consisting of world renowned experts in material characterization. Our staff, led by team conducts collaborative research projects with leading material research labs around the world. They regularly publish articles in leading peer reviewed journals, and speak at technical symposiums around the world.

For almost half a century Quantachrome's scientists and engineers have revolutionized measurement techniques and designed instrumentation to enable the accurate, precise, and reliable characterization of powdered and porous materials. We have an unwavering commitment to providing state of the art technology, along with superior and unparalleled customer service and support.

Our commitment to customers is to support you before, during, and after the sale throughout the lifetime of our instruments. This is a big commitment because our products are so robust and reliable that we regularly find many still in use for decades.

## Corporate Headquarters-USA

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Materials and Powder  
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