

Catalysts



Ceramics



Energy



Carbon



Pharma



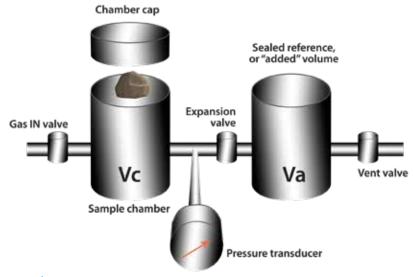
True density analysis of powders, foams and bulk solids.



# **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 any 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 inert behavior. Other gases, such as nitrogen, are also routinely used with no measureable 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. The pressure drop is then compared to the behavior of the system when a known volume standard underwent the same process.

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 accuracy of your measurement will suffer. This is why 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</li>

# True Density Analysis of Powders, Foams and Bulk Solids



# **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**.

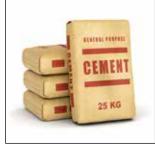
# **Dried Film Coatings**

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

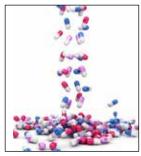
#### Metallurgy

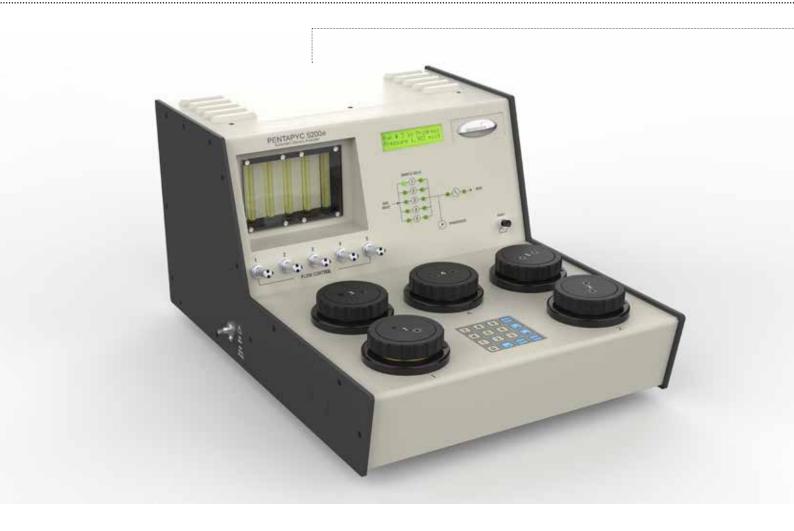
The true density of complex metal shapes formed by powder metallurgy is used to track physical properties throughout the processing of such structures. See MPIF Standard 63.











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. Its automatic repeat measurement mode ensures that 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. Connection and an RS232 balance interface. Complete with three inter-changeable 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

# 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 cm³)
   Volume Accuracy-better than +/- 0.02%
   Repeatability -better than +/-0.01%
- Medium Sample Cell (50 cm³)
   Volume Accuracy-better than +/- 0.03%
   Repeatability -better than +/ 0.015%
- Small Sample Cell (10 cm³)
   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 of 50% of the cell volume.

# **Special Versions**

# Foam Measurements

The 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).

### **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.

# **Automatic Gas Pycnometers**

**PentaPyc / PentaFoam 5200e** instruments from Quantachrome 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.

# Easy to Use

Samples are easy to load and unload. Measurement protocols are easily invoked at the unit or via a connected PC.

#### **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 of up to 5 samples frees the operator for other tasks while the unit works unattended.

# **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 – 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.

# Pre-Calibrated, Re-Calibrate Any Time

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

#### 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.

# Direct Input of Sample Weight

The PentaPyc/PentaFoam instruments can connect to an electronic balance to obtain the sample weight directly. Weight can also be entered at the keypad or via the PC.

# **Direct Operation**

Analyses can be set up and started using the built-in keypad or a PC.

## **Customizable Analysis Parameters**

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

### **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.

#### **Automatic Troubleshooting Alerts**

Loss of gas pressure, unstable pressure, and other fault conditions are automatically reported.

# True Density Analysis of Powders, Foams and Bulk Solids



# **Accessories**



# Non-Elutriating Sample Cells

Non-elutriating sample cells have perforated lids that prevent instrument contamination when working with fine powders such as carbon black, toner, or fly ash.

### **Gas Regulator**

A 2-stage gas regulator with CGA fitting and an isolation valve on the output with a 1/8 inch gas line connector is recommended. A suitable gas regulator assembly is available for purchase from your local Anton Paar salesperson.

# **Temperature Control Module**

A compact yet powerful thermoelectric heater/chiller circulates liquid to the heat exchange system for precise temperature control.



# Foam Sample Preparation 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 a knife and a circular punch. The Foam Sample Preparation Kit is supplied with the **PentaFoam 5200e** and can be purchased separately.

### Sample Cells and Adapter Sleeves

Durable, easy to clean stainless steel sample cells and adapter sleeves are provided with each pycnometer. Additional sizes and Delrin versions are available to best fit your specific application needs.



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: <a href="mailto:application-sp@anton-paar.com">application-sp@anton-paar.com</a>), 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.

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