The HumiPyc™ (Model 2) - Gas Pycnometer; Density, Moisture, Permeation Analyzer; Filter Integrity Tester; RH sensor Calibrator

Designed, built, and supported by InstruQuest Inc.

Universal pycnometer, no elutriation, lab and/or field (can use its own air supply system)- a replacement of air comparison pycnometer.

The HumiPyc, Model 2 is a fully automatic, versatile, precise, and cost effective volumetric analyzer capable of carrying out density measurements of different samples at ambient temperature. With ancillary equipment, it can be used for filter integrity testing, moisture analysis, permeation of gases through membranes, and calibration of RH sensors using saturated salt solutions.

Opposite to On/Off valves used in other pycnometers for pressurization / depressurization of sample chamber, the HumiPyc uses proportional valves to ensure continuous flow of gas in and out of the sample chamber, from vacuum to 345 kPa. Thus, elutriation of fine powders is avoided. Addition of RH probe to the sample chamber allows for density determination at known conditions of RH, temperature, and pressure. A unique design of the sample chamber closure enables variety of applications.

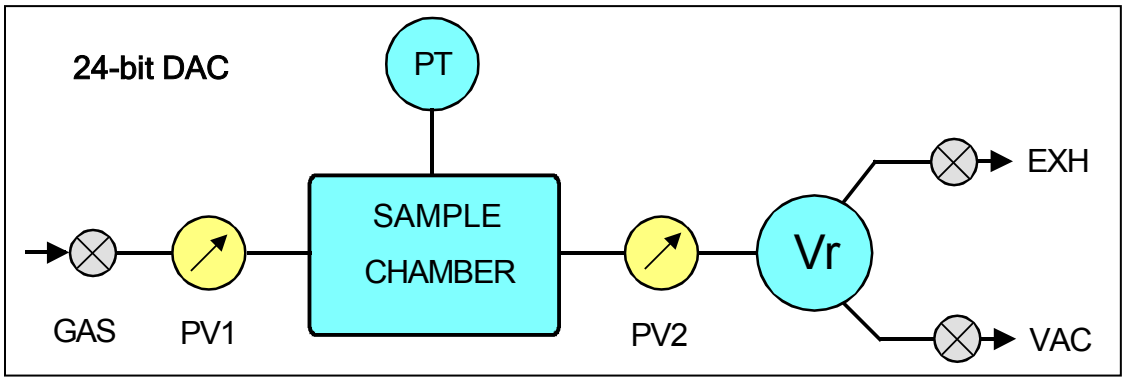
New analytical capabilities and convenience of operation are materialized thanks to migration from classical keypad operated pycnometers to PC environment for software design and control. A unique design of software allows the user to design the experiment as a sequence of pre-programmed steps (macros), and save the created template for future use. A new experiment can be executed with just a few clicks. Experimental parameters, if needed, can be modified during a run. All experimental data are recorded and can be transferred to a spreadsheet, e.g. Excel, for further analysis using supplied macro.

In addition to the Auto mode, a Manual mode is also available, mainly for troubleshooting and carrying out specific R&D testing of samples. The LCD displays all relevant data and a PC is not needed for this mode of manual control.

The instrument can be equipped with integral air supply system and a 12VDC power port mainly for field applications (geology, agriculture).

Applications

- True density measurements at precise conditions of RH, pressure, and ambient temperature without elutriation (fine powders)
- Determination of volume (density) versus used pressure value (open-, closed-cell foams)
- Filter integrity testing (Bubble Point and Pressure Decay methods)
- Calibration of RH sensors using saturated salt solutions
- Permeation of gases and vapors through membranes (optional equipment needed)
- Moisture analysis, desorption, headspace extraction, sample treatment



Simplified operational schematic of HumiPyc as a gas pycnometer

In the basic configuration of HumiPyc as a gas pycnometer, the carrier gas (typically helium or nitrogen) is delivered to the sample chamber by opening the GAS valve and controlling the voltage to the proportional valve PV1 to achieve the desired pressure value in a progressive way. Upon establishing a steady pressure reading from the transducer PT, and assuming prior establishing of ambient pressure in the reference chamber Vr, the second proportional valve PV2 is opened in a controlled way to allow for continuous transfer of gas from the sample chamber to the Vr. The exhaust valve, EXH, and the vacuum valve, VAC, are closed during this operation. The established pressure value after depressurization is read for subsequent calculations. The PV2 is also used in a similar way to either establish ambient pressure in the sample chamber, (EXH valve needs to be opened), or when connecting vacuum port to the sample chamber (VAC valve needs to be opened).

The continuous way of gas transfer into and out of the sample chamber eliminates the elutriation problem of fine powders. Additionally, there is no need for a special design of (expensive) sample containers or sample chamber to alleviate this problem. As a benefit of the design, the user can utilize any sample container that can fit into the chamber. Also, since the flow control allows pressure buildup to any user defined value, the influence of the pressurization pressure on the sample volume can be easily obtained by programming a series of pressure steps in ascending and/or descending order. This is particularly useful for determination of density of foams and other easily compressible materials.

An excellent temperature uniformity and reliability of operation is achieved by machining the whole measuring system from a solid aluminum block. As a result, the number of fittings is minimized and the chances of leaks are substantially reduced. The temperature of the aluminum block is measured by RTD based temperature probe (RTD1). Additional (optional) temperature probe (RTD2) is available for additional applications.

Another unique feature of the HumiPyc is **the ability to monitor relative humidity (RH) and temperature of the headspace over the sample** by using RH probe. Since the level of RH can reduced to a desired level by gas cycling or use of vacuum, the density determination can be carried out at a predefined level. This feature allows for better understanding of moisture factor in density variations between samples as well as observing trends of density changes with changing levels of moisture content.

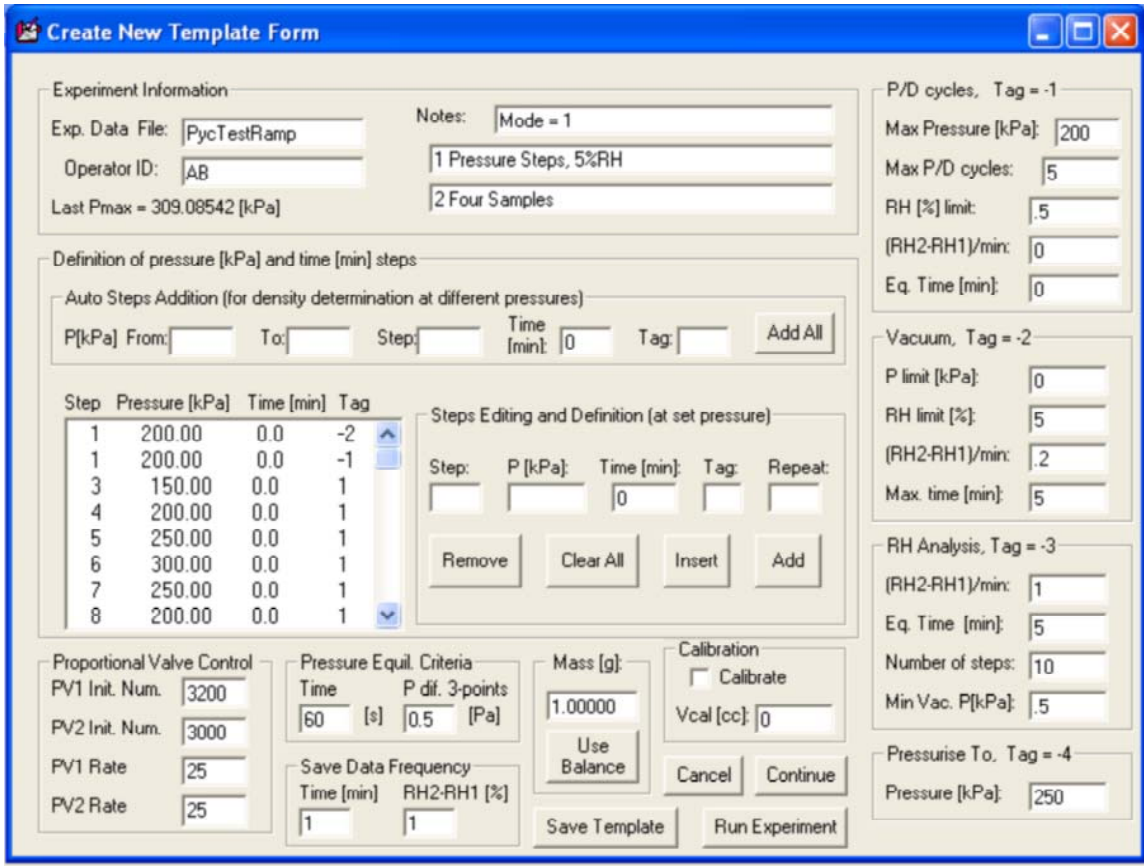
The sample and reference chambers and all passages are machined from a solid aluminum block. Since the valves are mounted in the block, from the entrance of the gas to the exhaust there are no fittings that can be a source of leaks. Therefore, the design increases reliability of operation. A proprietary design of sample chamber closure allows usage of different modules, with or without sensors. In cases of having only a small sample amount, the void volume can be reduced by using one of the supplied adapters.

One of the design objectives was to combine dedicated manually operated pycnometers (foam density, closed-, open-cell) and automatically operated ones (keypad) into a single design where the full capabilities

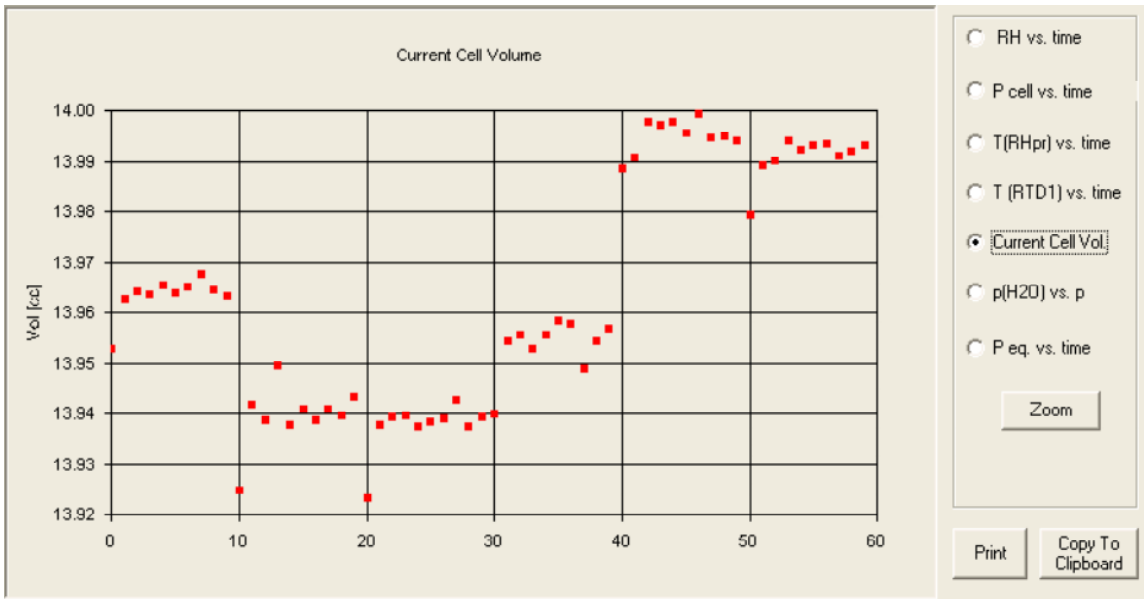
of the volumetric system can be materialized in the Windows® based software environment. The Auto mode of operation is controlled via intuitive and interactive software that is supplied with the instrument. A USB or a serial port of the PC can be used for communication with the instrument.

Experiment design and control using Windows® based PC software

User-friendly software for execution of different RH steps, diagnostics, calibration, graphing, reporting, and RH calculations has been designed. The user can conveniently define an experiment as a "sequence of steps" using the template form and save it for future use. A single step can be a complete measurement cycle, a sample treatment, or some special functionality. Predefined routines containing user modifiable parameters are assigned a tag number, which can be introduced to the sequence of the experiment steps. Automation of templates design, flexibility in experiment modification during run, running multiple samples using the same template, and ability to switch between Auto and Manual operation are very useful in any research work. A snapshot of the template design screen is presented below.



During a run, the user has the ability to view data from multiple sensors and monitor equilibration profiles, volume changes vs. cycles, etc. As an illustration, a snapshot of the screen presenting volumes of sample chamber for several different samples, each with ten cycles (repetitions) is shown below. The operating software records experimental data in several files: one file contains every communication transaction with the HumiPyc, second contains all experimental data (*.DAT), and the third one being the summary (*.SUM) file with the extracted volumes values. A macro that allows transferring (and plotting) the data files into Excel is supplied with the software.



In addition to determination of volume and density of samples at defined RH, temperature, and pressure, the HumiPyc can be employed in further characterization of samples and other optional analytical techniques.

RH analysis - using vacuum and sequential extractions from the sample chamber using the reference chamber, desorption of moisture from samples and comparison of moisture holding ability among different sample can be made.

- using pressurization to a user defined pressure level and subsequent depressurization to the ambient pressure, the changes of RH can be monitored and comparison among samples can be made.

Filter integrity testing – Using specially designed adapters (transparent), the Bubble Point and Pressure Decay methods can be implemented for filter integrity testing.

Sample treatment – after subjecting the sample to a vacuum and connecting a source of vapor to the sample (manual mode), the changes in density and desorption of the substance from the sample can be evaluated.

RH sensor calibration – using saturated salt solutions, RH sensor calibration can be carried out form vacuum to maximum allowable pressure and at different temperatures.

Permeation of gases and vapors through membranes – using additional optional hardware, rates of transport of gases and vapors through membranes can be measured. Using vacuum on one side of the membrane while the other side is at ambient pressure, or using some above pressure value while the other side is at ambient (or vacuum), the measurements of transport rates can be carried out. Using the pressure gradient method, much faster samples turnaround can be achieved. Introducing a known level of RH to the carrier gas allows for studying dependence of transport rates on the level of RH.

HumiPyc Model 2 Specifications:

Analytical techniques: **GAS (Helium) PYCNOMETER -** Volume measurements from under 1cc to over 100 cc (true density) of solids (fine powders, foams), optionally at precise RH conditions.

OPTIONAL TECHNIQUES: Filter integrity testing using Bubble Point and Pressure Decay Methods; **Permeation** testing using pressure gradient method

Operational mode: Fully automatic operation via PC control (Windows® based software from 95 to Windows 7 is included) and Manual Mode via front panel controls.

Maximum number of cycles per run: 1000 (with a single mouse click to continue another experiment)

Data handling: Printable reports, all data are recorded and transferable to spreadsheets

Number of reference chambers: 2 (small and large)

Sample chamber: Typical chamber volume is over 130 cc, larger volumes available, several adapters for reducing volume are supplied.

Sample containers: Several containers of different sizes are supplied, no special containers are required. Commonly available containers that can fit inside can be used.

Sample treatment: Sample treatment to specified criteria; programmable and continuous pressurization/depressurization cycles or vacuum. Optional **true purge** with flow through the sample, (not around the sample container like in other pycnometers), is available.

Volume calibration: Using certified metal spheres, Calibration kit is included
(Set of Large spheres (0.5" -2"), Set of micro spheres (1 – 6mm), handling tools)

Typical Accuracy: Better than ±0.03%

Typical Reproducibility: Better than ± 0.015%

Resolution of data acquisition: 24-bits

Volume resolution: Better than 0.0001 cc

Flow/Evacuation rate: Progressive, user programmable, (Fine powder sample is not blown out as proportional valves are used instead of ON/OFF valves for critical operations).

Pressure range: (transducer dependent) typically 344.7 kPa (50 psia), absolute

Displayed pressure resolution: 0.0001kPa

Transducer selection: Absolute, Gauge, Barometric, (common ranges)

Transducer accuracy: (transducer dependent), typically ±0.11%FS, ±0.073%FS optional

Temperature: ambient

Temperature probe (RTD) accuracy: ±0.1 ˚C

RH probe range: 0 to 100 %

Pressure regulation: Built-in low pressure regulator (up to 20 bar (300 psig) input limit) and pressure gauge at the back panel

Gas Type: Helium, Nitrogen, Argon, air, etc

Gas Inlet Port: 1/8" tube compression tubing (Swagelok® type bulkhead)

Vacuum port: 1/4"NPT Female (standard), flexible vacuum hose from the instrument to a vacuum pump (e.g. small rotary vane) with KF16 flange can be supplied.

Auxiliary hardware: Specific to an optional technique or customized version.

Communication link with a PC: USB or Serial port (RS232)

Dimensions: (W x H x D) (22 x 28 x 35cm) (8.7" x 11.4" x 13.7")
(Not including protrusions at the back)

Instrument Weight (option dependent, w/o accessories): About 7 kg (15 lb)

Typical power requirements: (Depending on specific model)
110-240 VAC universal input, 50/60 Hz, 75VA

These specifications are subject to change at any time and are dependent on specific versions.

Note: Performance of pycnometers varies with selected experimental conditions and hardware, (please review the posted application note)

InstruQuest Inc.
P.O. Box 970366
Coconut Creek, FL 33428, USA

Ph: (561) 271-1958
Fax: (561) 477-8021

E-mail: ***info@instruquest.com***
www.instruquest.com

Distributed by: