The HumiPyc™ - Model 1 - Gas Pycnometer; Density, Moisture, Permeation Analyzer; RH sensor Calibrator

Designed, built, and supported by InstruQuest Inc.

Temperature controlled, multi-technique volumetric analyzer, no elutriation

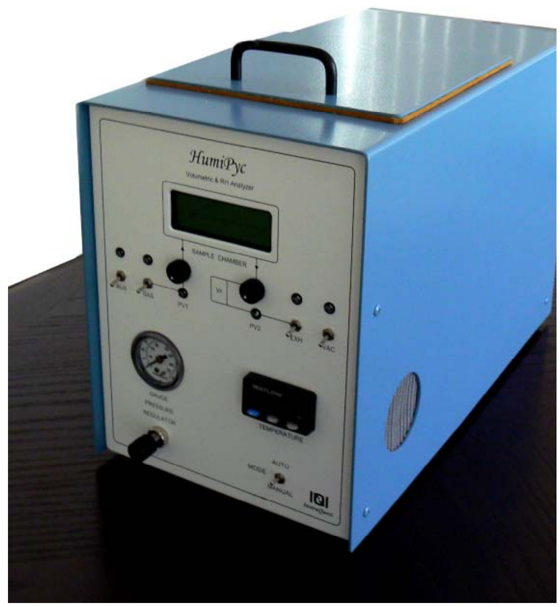
The HumiPyc is a fully automatic, versatile, precise, and cost effective volumetric analyzer capable of carrying out density measurements, moisture analysis, permeation of gases through membranes, and calibration of RH sensors using saturated salt solutions.

The temperature control from sub-ambient up to 50 ˚C allows for measurements at a well-known and uniform temperature. Opposite to On/Off valves used in other pycnometers, the HumiPyc employs 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 defined conditions of RH, temperature, and pressure.

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.

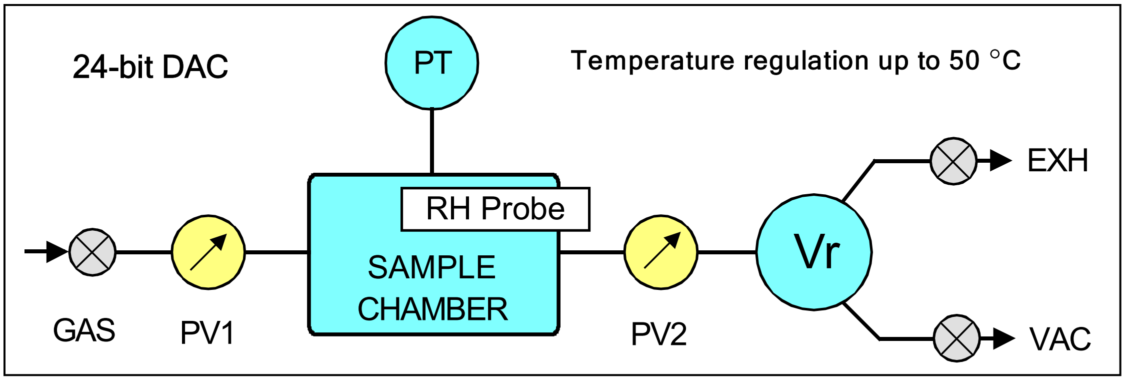
In addition to the Auto mode, a Manual mode is also available, mainly for troubleshooting and carrying out specific R&D testing of samples. The LCD displays all relevant data and a PC is not needed for this mode of manual control.

The instrument is equipped with a non-relieving type precision pressure regulator and pressure gauge. Since it isolates the instrument from fluctuations in the supply gas line, it can be connected to any common gas source and there is no need for a dedicated gas supply.



Applications

- True density measurements at precise conditions of temperature, RH, and pressure, no elutriation (fine powders)
- Determination of volume (density) versus used pressure value (open-, closed-cell foams, etc)
- 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 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.

Temperature control from sub-ambient to up to 50 ˚C and excellent temperature uniformity and stability allows determination of a sample volume at a defined temperature and allows study of the sample volume changes when different temperatures are used. A Peltier based cooling module is employed in the temperature control system. The desired temperature is set manually on the Watlow temperature controller.

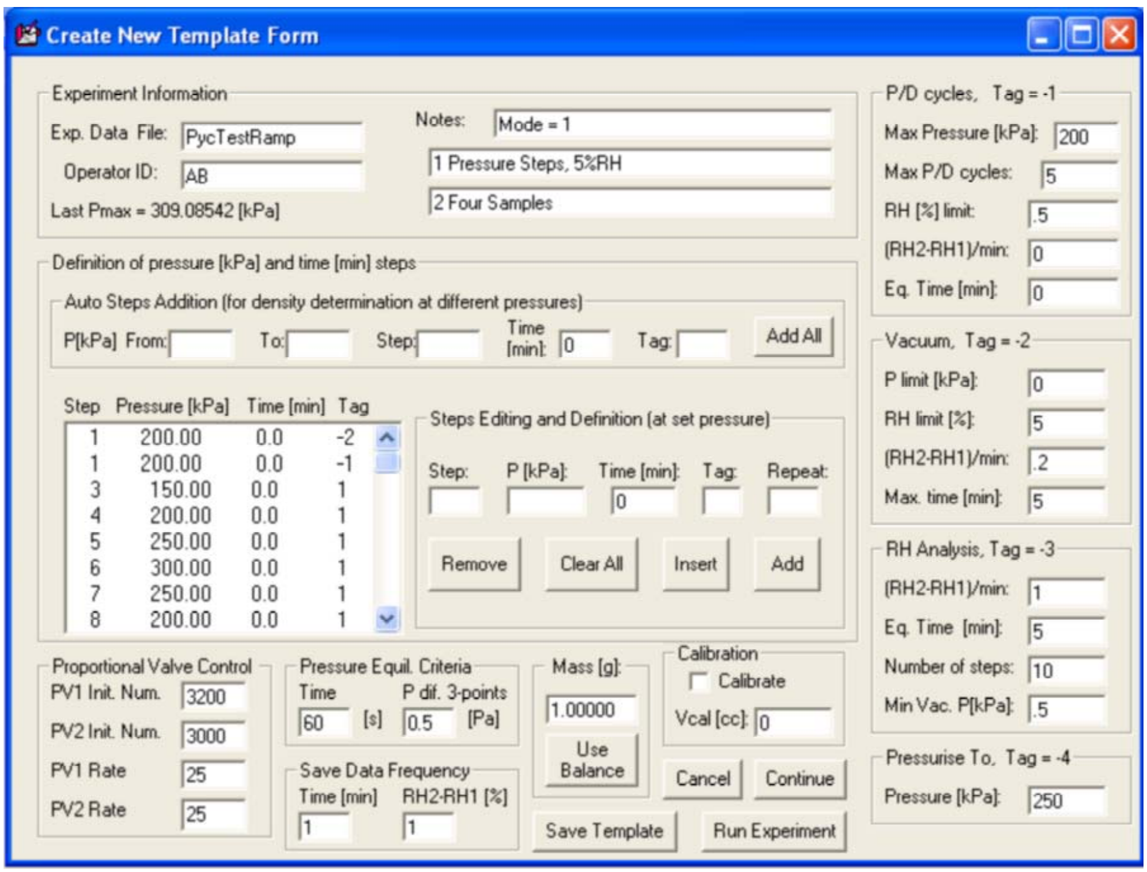
Another unique feature of the HumiPyc is **the ability to monitor relative humidity (RH) and temperature of the headspace over the sample** by using RH probe. Since the level of RH can reduced to a desired level by gas cycling or use of vacuum, the density determination can be carried out at a predefined 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.

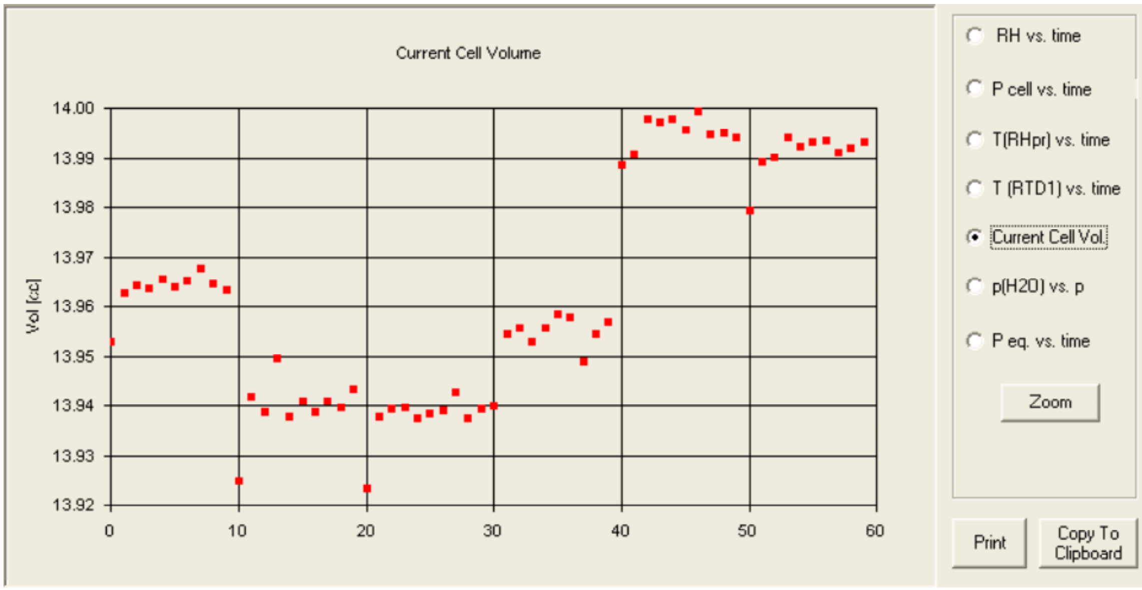
One of the design objectives was to combine dedicated manually operated pycnometers (foam density, closed-, open-cell) and automatically operated ones (keypad) into a single design where the full capabilities of the volumetric system can be materialized in the Windows® based software environment. The 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.

Headspace extraction - passing the gas through the user implemented sample loop and subsequent injecting to a gas chromatograph would allow study of concentration changes of a desired substance.

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.

Study of RH over sample headspace – varying pressure and temperature, the RH changes of headspace over variety of substances can be determined.

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 moisture presence.

HumiPyc Model 1 Specifications:

Analytical techniques: Volume (density) determination at precise RH and temperature conditions (No-Elutriation), Relative Humidity Analysis, Multiple Headspace Extraction, (optional) permeation of gases and vapors through membranes)

Operational mode: Fully Automatic (Windows® based Software) and Manual Mode

Sample chamber: Maximum volume about 125 cc, (5.3cm OD x 5.7cm H) (2.1"OD x 2.25"H), several adapters for reducing volume are supplied

Sample containers: No special containers required, commonly available containers (with lids, caps) of standard sizes can be used, several different types are supplied.

Sample treatment: Sample treatment to specified criteria; programmable and continuous pressurization/depressurization cycles or vacuum; introduction of vapors (manual mode only).

Volume calibration: Using SS spheres
(Set of Large spheres (0.5" -2"), Set of micro spheres (1 – 4mm), optional set of medium spheres (1mm – 25 mm or 1/8"-1"), Grade 25 or better)

Detection limit (volume repeatability): <±0.010 mL

Standard deviation of repeatability: < 0.005

Resolution of data acquisition: 24 bit

Pressure mode: Continuous and rate programmable pressurization and depressurization of samples, programmable pressure steps

Pressure range: (transducer dependent), typically from vacuum to 50 psi (344.735 kPa),

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

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

Temperature range: sub ambient to 50 ˚C, Peltier cell(s) based cooling (no bath needed)

Temperature regulation: < ±0.1 ˚C

Temperature probe (RTD) accuracy: ±0.1 ˚C

Thermal protection: Programmed limits and thermal cut-offs.

RH probe range: 0 to 100 %

RH (digital) probe accuracy / resolution: ±1 %RH / 16 bit

Gas Type: Helium, $N_{2,}$ air, etc

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

Auxiliary supply ports: application specific connectivity

Gas Inlet Pressure:
Maximum: 150 psi (10.3 bar)
Minimum: 20 psi (1.4 bar) above the maximum pressure to be set on the instrument panel

Gas set point pressure: Precision pressure regulator (non-relieving) and gauge are integral to the instrument, front panel

Vacuum port: 1/4"NPT Female (standard), other adapters available

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

Dimensions: (W x H x D) (22 x 29 x 43cm) (8.7" x 11.4" x 19")
(Not including protrusions in front, back, and top)

Instrument Weight (w/o accessories):
14kg (30 lb)

Typical power requirements: (Dependent on specific model)
110/120 VAC, 300VA, 60 Hz nominal
(Optional): 220/240 VAC, 300VA, 50 Hz nominal

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

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

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