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Edu-Pyc™ 101 Educational Gas (Helium) Pycnometer Theory and Practice

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A gas (helium) pycnometer is used for determination of volume (density) of predominantly solid state samples (powders, foams, etc). Many automated models have been developed for either general use or for specialized applications and the popularity of pycnometers is increasing due to being indispensable tools in materials characterization. However, the instrumentation industry tends to create “well finished” but expensive models which are dedicated to the specific task only with more focus on how to operate the “black-box” through software then to provide solid understanding of the technique and its hardware.

The main objective of the introduction of the **Edu-Pyc™ 101** (cited later in the text without the trade mark sign) is to focus on educational and practical aspects of pycnometry by providing a simple hardware to gain hands-on experience, as well as achieve basic understanding of this method. It is highly recommended to follow and understand the theory as this simple application of the ideal gas law $pV = nRT$ is used in many much more complex volumetric instruments.



Fig. 1. Illustrative version of the Edu-Pyc 101 Pycnometer

The Edu-Pyc 101 is a manually operated gas pycnometer and it is provided as a complete unit. It is an open design with minimal hardware needed for carrying out experiments for volume (density) determination of samples. All the hardware is easily accessible to the user for easy learning and any modifications.

The 3-port manifold in the upper left corner is designated for connection of the gas supply and the gas pressure is indicated by the pressure gauge connected to this manifold. The typically installed fitting (Swagelok®, male connector, compression, 1/8” NPT) accepts 1/8” OD tubing but other types can be used if more suitable. From the 3-port manifold the gas is connected to the pressure regulator which is used for setting the required pressure level for a given experiment. After regulation the pressure is indicated by the

pressure gauge attached to the regulator. The pressure regulator output is connected to the leftmost (pressurization) On/Off toggle valve which allows pressurization of the reference chamber. The pressure established in the reference chamber is indicated by the digital pressure gauge (battery operated). The middle (expansion, depressurization) toggle valve (On/Off) connects the reference chamber to the sample chamber and allows expansion of gas if it is turned on (lever upward). The third toggle valve (exhaust) is connected directly to the sample chamber and allows for release of gas pressure to the atmospheric value. Although the toggle valves are of On/Off type, they can be of different design. For example, the lever of the leftmost valve is rotate-able, and allows flow only if the lever is brought to position along the stem of the valve.

To gain access to the sample chamber, the large knurled knob needs to be turned counter-clockwise sufficiently enough to allow the arm to be moved to the resting position, either forward or backward. Then the sample chamber cover can be lifted up. The provided sample holder with the hermetically closed cover is designed for holding the sample or the calibration ball. The magnetic pickup tool is provided for convenient insertion and removal of the calibration ball.



Fig. 2. Opened sample chamber with the sample holder placed inside.

The presented design can have many modifications and the users can introduce their own customization. Assuming mechanical integrity of the system (no leaks), the digital pressure gauge is the most decisive factor determining accuracy of measurements (and cost). The selection of the digital pressure gauge will depend on the type of application. Just for learning, a lower cost digital pressure gauge (pressure transducer) with accuracy of 0.25 % Full Scale (FS) or so can be used. For carrying out the more exacting measurements, transducers of accuracy equal or better than 0.1 % need to be employed but they can be quite expensive. Any pressure transducer/gauge with 1/4" NPT Male connection can be utilized.

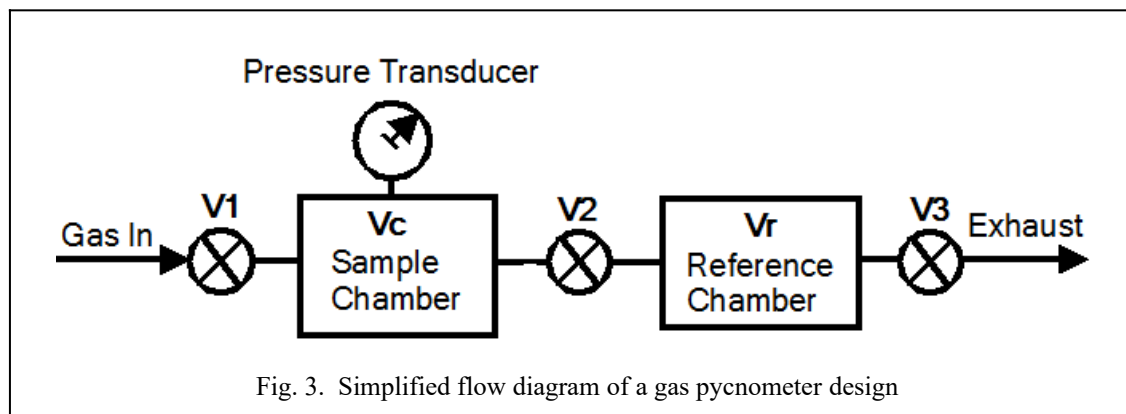
The automated gas pycnometers offer sophisticated software and convenience and are the preferred choice for QC applications when many runs per day are to be carried out. However, for occasional determination of volume (density) of samples, this pycnometer can be very handy, especially for field applications as there is no need for external electrical power and a small air compressor (or even a hand air pump) can serve as a gas source. Since there are no electrically actuated valves, there is no heat buildup caused by valves coils or leaks caused by low power electronic valves. The robust design can serve for decades of operation and the user can change or customize it as needed.

The heavy-duty sample holder can be hermetically closed if its cover has O-ring installed in it. A fritted SS filter is installed in the sample holder cover to reduce/eliminate escape of fine powders during pressure cycles. Filters of various porosity (2, 5, 10, 20, or 40 microns) can be used. The calibration ball, either 45 mm or 1.75", can easily fit inside the sample holder.

Basic Functionality and Theory

A gas (Helium) pycnometer is a laboratory or field instrument that measures volume (density) of materials using gas displacement method by employing the ideal gas law, $pV = nRT$. The name Helium pycnometer was used extensively in the eighties, mainly for the marketing purposes of new automated pycnometers where the speed of operation was the main feature against the common air comparison pycnometers at that time. Practically, any (neutral) gas that does not react with the sample can be used and for most samples the results are indistinguishable when using Helium or nitrogen.

The basic operation relies on having a user accessible sample chamber V_c , and an added volume (reference chamber) V_r , often more than one. There are usually at least three valves used: the first (V_1) provides gas to the sample chamber (Pressurization valve), the second (V_2) valve allows expansion of the gas from sample to reference chamber (Expansion or depressurization valve), and the third (V_3) releases the gas to atmosphere to establish atmospheric pressure P_a value (Exhaust valve).



There are also other designs, where the position of the sample and reference chambers are reversed, or connections of the valves can be different (e.g. the V_3 valve can be connected to V_c and exhaust is done through the sample chamber, not through the reference chamber as in the above diagram). Since usually one pressure transducer is being used, its connection to the sample chamber is preferred when additional analytical techniques are to be implemented by using the resources of the pycnometer and additional auxiliary hardware. Generally, pycnometers designs can be much more complicated, and the particular flow diagram should be studied to understand functionality of a given unit. It is important to know, that equations derived for the flow diagram as on Fig. 3 or for the reversed positions of V_c and V_r , are a bit different.

The volume of the sample chamber is user modifiable in a sense that various sample holders, samples, volume reducing adapters can be introduced into the sample chamber. The reference chamber volume is intended to remain constant and in most designs there is no direct or easy access to it. In general, the volumes of sample and reference chambers are initially unknown. Using an object of known volume, usually a high precision metal ball (sphere), the volume of the reference chamber can be determined through the so-called calibration process. Once the volume of the reference chamber is known, the volumes of the sample chamber, with the sample and without it, can be subsequently measured. The process is explained in more detail below.

The typical pycnometer sequence of operation consists of three steps:

1. **Opening of the V_1 valve, pressurization of sample chamber to the pressure P_p , and closing the V_1 valve.** After pressure has reached a stable value, the pressure P_p needs to be recorded.
2. **Expansion of the gas to the reference chamber by opening the valve V_2 (V_3 and V_1 stay closed).** After the depressurization, the same pressure P_d will be in both chambers, V_c and V_r . It is a good practice to temporarily open and close the V_3 valve to make sure that only the atmospheric pressure is

inside the reference chamber before the V2 valve is opened. Once the pressure value has reached a stable value Pd, the valve V2 can be closed, and the value of Pp needs to be recorded.

- 3. Release of gas to the atmosphere by opening valves V3 and V2.** The transducer will read the atmospheric pressure, Pa. If using an absolute pressure transducer, the recorded value will be close to atmospheric pressure (standard value is 101.325 kPa at sea level) but for so called gauge type transducers, the Pa value will be close to 0 kPa.

The above set of valves operation consisting of the pressurization, depressurization (expansion), and gas release, and recording pressures Pp, Pd, and Pa is used throughout each experimental stage of volume determination or calibration. Such cycle can be repeated many times to obtain a statistics of repeatability. It should be clearly understood, that the standard deviation of such statistics is not a measure of actual accuracy of volume measurements as in automated pycnometers the repeatability can be very good, but the actual results can be far off in case of a malfunctioning pycnometer.

Let us carry out the first experiment with “empty” sample chamber (meaning no sample or no calibration ball, but a suitable sample holder or other hardware can be placed inside). When the sample chamber is pressurized, the number of moles of gas in it is $P_{p1} \cdot V_c / (R \cdot T_c)$. At that time the number of moles in the reference chamber being at atmospheric pressure is $P_{a1} \cdot V_r / (R \cdot T_r)$. After expansion and establishing the same pressure in both chambers Pd1, the total amount of gas is preserved. It is assumed, that the temperature during the pressurization and depressurization (expansion) remains the same and the temperature of the sample chamber and reference chambers are the same $T_c = T_r = T$. So the mass equation can be written as:

$$\frac{P_{p1} \cdot V_c}{R \cdot T} + \frac{P_{a1} \cdot V_r}{R \cdot T} = \frac{P_{d1} \cdot (V_c + V_r)}{R \cdot T} \quad \text{Eq. 1}$$

The subscript 1 is used to denote the set of pressures in the first experiment and consecutive values are used in the next experiments (cycles). After simple manipulations, the above formula can be rearranged as:

$$V_c = V_r \cdot \frac{P_{d1} - P_{a1}}{P_{p1} - P_{d1}} \quad \text{Eq. 2}$$

It is clear from the equation 2, that one experiment in which the three pressure values are known, provides only a relationship between Vc and Vr. and that further work is needed to determine one of the volumes. The strategy is to change volume of one of the chambers by a known value. Let us reduce the volume of the sample chamber as there is an easy access to it by inserting an object of known volume, Vobj, and carry out the second experiment using the same experimental steps and writing the second mass conservation equation. Out of convenience, availability, and ease of measurements, the object is usually a precision metal or ceramic ball (sphere), but a cylinder or cubic shape pieces can be used as well.

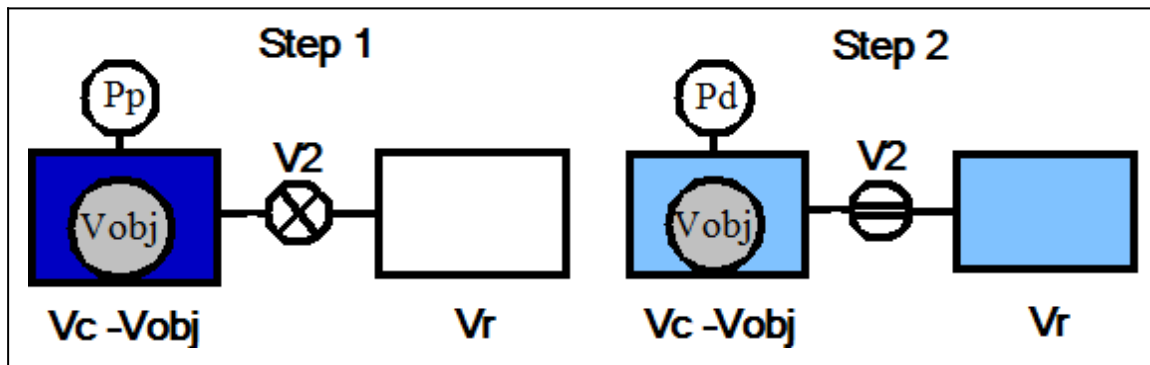


Fig. 4. Calibration: Step 1 – pressurization, and step 2 – expansion

This methodology is called the calibration of the reference chamber and the diagram above illustrates the process. Once the object of known volume is inserted into the sample chamber, its volume gets reduced from the initial V_c value to $V_c - V_{obj}$. Subsequent considerations are the same as in the first experiment.

$$\frac{P_{p2} \cdot (V_c - V_{obj})}{RT} + \frac{P_{a2} \cdot V_r}{RT} = \frac{P_{d2} \cdot (V_c - V_{obj} + V_r)}{RT} \quad \text{Eq. (3)}$$

The equation 3 states, that the amount of gas (number of moles) in the reduced sample chamber volume by the object volume at the pressure P_{p2} and the amount of gas at atmospheric pressure P_{a2} in the V_r volume must be equal to the amount of gas in both chambers after expansion to the pressure P_{d2} .

Assuming further constancy of temperature during the experiment, and substituting the V_c value from eq. 2 into the eq. 3, the V_r volume can be easily calculated as follows:

$$V_r = \frac{V_{obj}}{\frac{P_{d1} - P_{a1}}{P_{p1} - P_{d1}} - \frac{P_{d2} - P_{a2}}{P_{p2} - P_{d2}}} \quad \text{Eq. 4}$$

Once the calibration of the reference chamber is done, the measurements of sample volume are now very simple. Introducing the sample holder without sample into the sample chamber, and repeating the pressurization, depressurization, and release of gas to atmospheric pressure, the third set of the three pressure values is obtained and the V_c volume can be calculated as follows:

$$V_c(\text{w/o sample}) = V_r \cdot \frac{P_{d3} - P_{a3}}{P_{p3} - P_{d3}} \quad \text{Eq. 5}$$

Next, the sample amount is weighed, its mass recorded, and the sample holder with the sample is introduced back into the sample chamber. The results of the fourth experiment are calculated similarly:

$$V_c(\text{w/ sample}) = V_r \cdot \frac{P_{d4} - P_{a4}}{P_{p4} - P_{d4}} \quad \text{Eq. 6}$$

The sample volume, V_{sample} is the absolute difference between the eq. 5 and 6.

$$V_{\text{sample}} = | V_c(\text{w/o sample}) - V_c(\text{w/ sample}) | \quad \text{Eq. 7}$$

The absolute value is used as the sequence of the experiment without the sample and with the sample can be reversed. Finally, knowing the sample mass m , its density d can be calculated using the definition

$$d [\text{mass/volume}] = \frac{m}{V_{\text{sample}}} \quad \text{Eq. 8}$$

As always, a theory should be followed by some exercises to help better understanding and applying it in practice. Please try to solve the following problems by yourself.

Problem 1. In case you think that you have understood this simple theory, here is an easy problem to solve. The diagram below shows two hypothetical runs, where each consists of several repetitions of the basic cycles (pressurization, expansion, and releasing gas to atmosphere). The measured pressure values are presented as red and green circles. Can you tell (with explanation) which run is done with the sample and which is done without the sample in the sample chamber, assuming everything else remains the same?

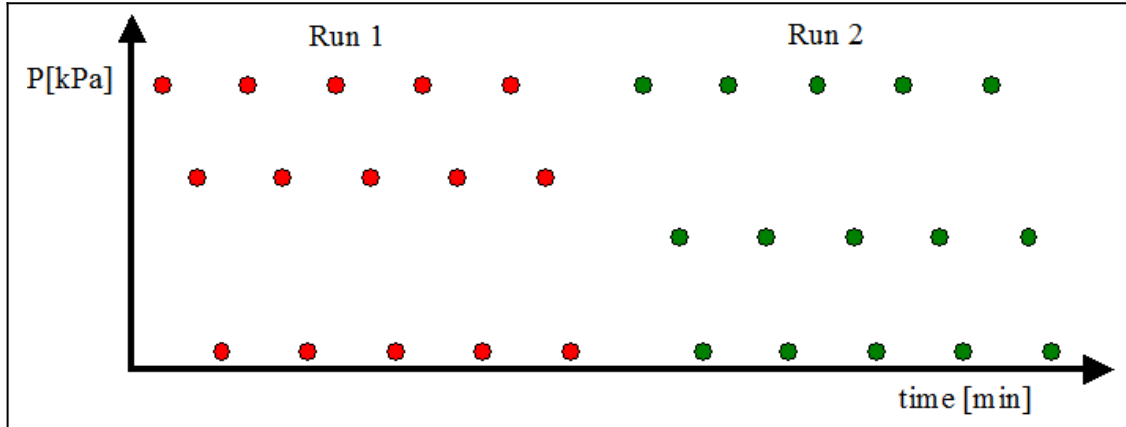


Fig 5. Graphical representation of several cycles repetitions

Problem 2. The above formulas derivations (Eq.1 through Eq. 6) were based on the design presented in the Fig. 3 where the sample chamber is initially pressurized. It is likely, however, that the instrument you will be working with is of a bit different design where the position of sample and reference chambers is reversed and the reference chamber is initially pressurized. Therefore, please derive the corresponding formulas for such case (and they will be really needed).

Problem 3. (Optional). In the above derivations, it was assumed that the sample and reference chambers are at the same temperature. But if they were physically separated and each kept at a different temperature, derive the formulas for such case.

Problem 4. (Optional). There are three samples available: powder A, powder B, and a mixture of them of unknown mass ratio. Having a gas pycnometer and a laboratory balance available, come up with a strategy (and formula) of how to find out the ratio of mass A and mass B in the mixture of the powders A and B.

Suggested experiments and calculations

1. Calibration of the reference chamber.

Comment	Pp [kPa]	Pd[kPa]	Pa[kPa]
Run 1, without Calibration Ball			
Run 2, with Calibration ball			

Task: Determine the volume of the calibration ball and calculate Vr

$$V_{\text{ball}} = \frac{4 \cdot \pi \cdot r^3}{3} = \frac{\pi \cdot d^3}{6} \quad \text{where } r \text{ is the radius and } d \text{ is the ball diameter} \quad \text{Eq. 9}$$

Before calculating Vr, the mass equations must be stated and solved for a particular pycnometer design first. If the sample chamber is pressurized first, then use eq. 4. In the other case use the following equation:

$$V_r = \frac{V_{obj}}{\frac{P_{p1} - P_{d1}}{P_{d1} - P_{a1}} - \frac{P_{p2} - P_{d2}}{P_{d2} - P_{a2}}} \quad \text{Eq. 10}$$

2. Determination of a sample volume or rechecking the calibration ball volume.

Comment	Pp [kPa]	Pd[kPa]	Pa[kPa]
Run 3, without sample or ball			
Run 4, with sample or ball			

Task: Determination of the sample volume or using the calibration ball as a sample and see if the obtained ball volume agrees with the calculated (geometrical) volume.

If in your instrument the sample chamber is initially pressurized, then use equations 5 and 6. In the opposite case when the reference chamber is pressurized, use the following equations:

$$V_c(\text{w/o sample}) = V_r \cdot \frac{P_{p3} - P_{d3}}{P_{d3} - P_{a3}} \quad \text{Eq. 11}$$

$$V_c(\text{w/ sample}) = V_r \cdot \frac{P_{p4} - P_{d4}}{P_{d4} - P_{a4}} \quad \text{Eq. 12}$$

The purpose of using the calibration ball as a sample is to find out if ball volume obtained from the pycnometric measurements is the same or being very close to the calculated volume from its diameter measurement (geometrical volume). This “reality check” should be carried out using the calibration ball (or a different one) to determine if the pycnometer functions properly or if there is a problem somewhere. Calibration results and its verification should be recorded and kept for future references.

3. Porosity, bulk and tapped density.

In the area of characterization of powders and granular materials there are other parameters used, like bulk density, tapped density, porosity, and others. Bulk density is defined as the dry mass of such material in a given volume. Such density is easy to determine using the provided sample holder, which has a flat bottom. Using a caliper, the distance from the bottom to the edge can be easily found as well as the inner diameter of the sample holder. After pouring in some amount of sample, forming a flat surface, and measuring the distance from the sample surface to the edge of the sample holder, the height of the sample bed can be determined.

$$V_{bed} = \frac{\pi \cdot d^2 \cdot h}{4} \quad \text{Eq. 13}$$

where d is the inner diameter of the sample holder and h is the height of the sample bed. The sample can be either previously weighed or the sample holder can be weighed before adding the sample and after. The V_{bed} is basically the geometrical (physical) volume occupied by a known mass of the sample.

The powder sample "as poured" can be compacted somewhat by using a mechanical tapper and such density is called the tapped density. The tapped density is achieved by mechanically tapping a measuring cylinder containing a powder sample according to some specifications, like number of strokes, their frequency, and height of the drop of each stroke. After observing the initial volume, the tapping reduces the volume to a fairly stable value. It is perhaps far more effective to apply a suitable vibrator at the bottom of the sample holder and finger pressed any suitable tool with flat surface covering the area of the sample

holder to form a flat bed of powder. This will reduce the height of the sample to a stable value in a very short time.

The porosity ϵ is a dimensionless quantity and is defined as the ratio of void volume V_v to geometrical volume V_g . If multiplied by 100, it is expressed as % of V_v/V_g . The void volume is the difference between the geometrical volume and the volume obtained by pycnometer.

$$\epsilon = \frac{V_v}{V_g} = \frac{V_g - V_p}{V_g} = 1 - \frac{V_p}{V_g} \quad \text{Eq. 14}$$

It is instructive to determine porosity of various porous materials. For example, in case of cementitious materials, the porosity is usually below or about 0.5, for sandstones the range is roughly from 0.07 to 0.30, while for some open-cell foams it can be a very small fraction, as the volume seen by gas is very small compared to the geometrical volume.

4. Compressibility

One can get impression that density is a single number used in characterization of a given material. Generally it is not the case as many real samples do not fulfill many tacit assumptions in the pycnometry technique. One of the assumptions is that the sample volume does not change its volume versus pressure, which is not fulfilled in case of easily deformable materials like plastic foams or fluffy powders. Even at a fixed pressure the volume of such material can slowly get compacted over time. So in general, the density of compressible materials cannot be described by a single number but rather by a curve of volume vs pressure, with the time factor involved. Such analysis is rather more suitable when using fully automated and specialized pycnometers but even by using this manual pycnometer the compressibility of samples can be studied.

One way is to set a few different pressures using the pressure regulator and carrying out volume measurements of an easily compressible material. This should yield different volumes for the same mass of the sample. Alternatively, a relatively high pressure can be initially set for pressurization. In the following few runs, only the depressurization (expansion) and release of the gas to atmosphere needs to be carried out. Since the previous pressurization and expansion pressures will quickly approach atmospheric pressure, the measured volume will approach a more stable value measured at low overpressures, as required by standard methods for foam measurements (about 20 kPa above atmospheric pressure).

5. Open-cell and closed-cell percentage determination

Many cellular materials, e.g. plastic foams can be characterized by their opened or closed cell contents. The general approach is to measure the volume of the whole sample, and subsequently cut the sample into pieces to expose the closed cell. Measuring the volume again and taking corrections for the open cells, it is possible to estimate the percentages of the open-cell and closed cells. In order to achieve satisfactory results, a relatively large sample needs to be used and more importantly, specialized machinery has to be involved to cut precise pieces of foam sample to obtain a well defined geometrical volume. Due to this issue, this methodology is only mentioned here.

6. Porous materials of open and closed pores structure

Any porous material can have open pores and closed pores. The closed pores are generally not accessible to the gas but any permeation (diffusion) into and out of the pores cannot be excluded. When measuring volume (density) of a bulk sample of such material, we obtain its volume by whatever space the gas can access, within the allotted time and pressures used. The bulk sample can be crushed into very small pieces and the volume can be remeasured. Converting it even to a fine powder and remeasuring its volume can provide some idea if the sample has closed pores and at what stage most of the pores are accessible to the gas used.

It needs to be mentioned, that in the area of porous materials characterization, there is a long list of density definitions (true, apparent, skeletal, envelope, absolute, ...), which are often confusing and depending on the source, they can be defined differently. So using a common sense is a better approach and the volume (density) obtained by using a gas pycnometer is such as what the gas can penetrate. The volume (density) values can be influenced by the sample nature, sample treatment, its history, particles size reduction, experimental conditions (pressure, temperature, gas type, equilibration time, ...), etc.

Practical considerations

The obtained volume V_r is the primary result of the calibration procedure. Typically the reference chamber is most often not accessible to the user or at least not changed during experiment and in this sense it can be considered as an invariant. However, that does not mean that a calibration done one time is good forever. The V_r volume is not directly measured like a ball diameter with a micrometer, but is measured indirectly by gas and the whole electronics is involved. Due to many factors involved, even consecutive calibrations of V_r will produce close but not exactly the same numbers. Calibration of V_r should be carried out when experimental conditions change, like a large change of pressurization pressure, change of gas, change of ambient temperature by a few °C, different hardware used, etc... It does not mean though that it has to be carried out for every sample volume measurement. The periodic calibration results should be recorded and kept on file for reference, as increasing or large discrepancies would indicate a developing problem. Using the calibration object as a sample, the obtained volume by pycnometer should be compared to the calculated volume. Such results should be added to the record of calibration. It is often a good idea to carry out a "dummy" experiment just to exercise the valves and transducer before actual calibration of V_r . When an instrument has not been used for a while and especially if equipped with a capacitive type pressure transducer, it can take more than just one "dummy" experiment needed to bring the functionality back to normal.

Looking at the form of equations 4, 5, 6, 10, 11, and 12, it is easy to notice that if the pressure value P is substituted by a linear relationship of pressure versus voltage v (a denotes slope and b denotes intercept),

$$P = a \cdot v + b \quad \text{Eq. 15}$$

then instead of pressure values in the equations, the voltage v can be used directly. That stipulates, that only linearity of voltage versus pressure is needed, not necessarily that the pressure transducer has to show the exact pressure value or that it needs to be zeroed. It is also not relevant what pressure units are used as long as they are used consistently for the calculations but such units should be selected as to obtain as many as possible non-zero digits on the LCD display (if using such one).

From the point of view of numerical analysis, the form of the equations employed for calculations is not favorable as sub-results are differences of two relatively large numbers which can yield a small number. Dividing the results of subtractions and subtracting them again can lead to relatively large errors. This has some consequences for the pycnometer design which requires higher quality components, especially the pressure transducer and the accompanying electronics. Usually a compromise needs to be found between the tendency of using higher pressures and what the rest of the mechanical system, especially low power valves, can tolerate. The appropriate ratio of the sample to reference chamber volumes and the sample amount should be used to spread the differences between the P_p and P_d , and P_d and P_a more or less equally. This defines some optimal range of pressures and sample volumes for the most accurate determination of sample volumes for a particular design. This is the main reason of more complex designs in order to accommodate larger range of sample volume measurements.

Specifications of accuracy is rather a complex topic and it is subjected to interpretations. On one hand there are often "marketing" values specified but the reality is usually a bit different. Perhaps the best approach is to carry out several measurements with empty sample holder and perhaps repeat them with a precision metal ball that is relatively close to the sample volume. The usage of such ball reduces (eliminates) effects

of any real sample. Having the two sets of data, one can estimate the spread of results and calculate the standard deviations. Such experimentation can be repeated on another day to have an idea about the range of fluctuations.

Let say that the spread of results of the set of repetitions is within 0.03 mL. Since *a priori* it is not known what volume of the sample will be used, this error is usually is related to the maximum volume of the sample chamber. Assuming that such volume is 120 mL, the relative error can be calculated as $0.03/120 \times 100$ which amounts to 0.025 % (quite a good number). However, the sample holder occupies some volume, there is always a dead space, and the sample volume is usually less than whole space of sample holder available, and also the sample volume is less than the volume it occupies due to its porosity. Let say that the sample volume is 30 mL, so $0.03/30 \times 100$ yields 0.10% error (a reasonable result). However, if only 1 mL of sample is used, then the relative error is 3%. So it can be easily concluded, that it is better, whenever possible, to use relatively a large amount of sample in large chambers to reduce the amount of relative error. Since for smaller chambers the spread of results is generally smaller, most pycnometers offer dual reference chambers and reducing adapters for the sample chamber to increase the range of volume measurements with satisfactory accuracy.

Another way of estimating any pycnometer performance at selected experimental conditions is to run a series of precision balls of known volumes (from small to large) as samples and compare the pycnometer results with the known volumes. That should provide a more realistic expectations as to the actual accuracy. If the pressurization pressure can be build almost to the same value each time, then the standard deviation of repeatability can be quite small. It is usually smaller than the actual accuracy and perhaps since it looks better on paper, it is wrongly used as accuracy of volume measurements in reports. One should keep in mind that there is always some non-zero error generated in finding the V_r volume during calibration and this contributes to the error generated during sample volume measurements.

There are also many assumptions that one should be aware of. In addition to the ideal gas behavior and perfect mechanical integrity of the instrument (no leaks, linearity of transducer), it is assumed that the sample is not easily deforming, the gas absorbed during pressurization is given off during expansion, no net chemical reaction with the gas used, no sample removal (elutriating) during pressure changes. Pressure cycles of dry gas remove moisture from samples and that can affect the sample state. Real samples rarely fulfill all the assumptions. When comparing the volume (density) results to literature data, be sure to review the experimental conditions if they are provided. Using different gases, pressurization pressures, equilibration times, sample amount, preparation, and its history, etc., may affect the results. Although high end pressure transducers have satisfactory temperature compensation features, their performance is affected by ambient temperature changes.

Keep in mind that a gas pycnometer measures only volume of samples and it should be calibrated for volume only. Be aware of efforts by some creative sellers who try selling unnecessary hardware or "standard samples" that are calibrated for density. Density involves measurement of volume (e.g. by pycnometer) and mass (by balance). The overall error of density calculation is a combination of errors from volume measurement and from mass measurement, as two completely independent pieces of equipment are involved. There is absolutely no need for any samples of "standard density". If ever a public tender is involved, provide only the generic requirements for a pycnometer which you really need, not any specific features for a particular model and any additional (but often not needed) hardware or services that the seller is trying to include as the overall cost will be very high. Perhaps the most decisive factor in selecting a pycnometer is the planned sample volume to be measured (if powder) or its geometrical dimensions (irregular solids) as it needs to fit into the sample holder and the range of volumes that will be measured, in addition to performance, automation (software), cost, etc.

There are many samples that have characteristics between solids and liquids, e.g. glues, pastes, slurries, etc. Density meters that are designed for liquids only cannot measure such substances. Also, it can be too difficult to fill uniformly specialized glassware pycnometers designed for volumetric determination of density. Using gas pycnometers seems to be the last resort, but it can be also problematic considering some of the assumptions listed above. The practical problem with cleaning can be easily solved by using

disposable plastic bags that fit into the special design sample holders with covers containing metal filters to reduce pressure changes and practically eliminate sample escape.

There are also substances, like silica fumes, that are highly volatile, easily agitated by pressure changes, and can pass through fine filters. Since such substances can escape the sample chamber during pressure release to atmosphere, the sample holder with such sample should be weighed before and after experiment to check for any mass loss. The practice of sample weighing (or the sample holder with the sample) before and after experiment is recommended for any sample. When working with highly volatile samples, it is important to ensure safety measures. Perhaps the best way is to connect a flexible tubing to the exhaust port and redirect it either to the lab hood or outside of the room to avoid accumulation of the dust in the air. The same safety precautions should be practiced when dealing with unknown samples.

Even such a simple pycnometer like the Edu-Pyc 101 can be utilized for other purposes as some hardware resources, like the pressure regulation and pressure transducer can be a part of other setups. Additional ports in the sample and reference chambers are included. One of the attractive applications is usage of pycnometers for studying of gas flow resistance through permeable barriers, like packed beds or rock cores, essentially as a gas (air) permeameter. Connecting an external cell holding the packed bed to the pycnometer, and using a bubble meter and stopwatch, flow rates through packed beds of various materials can be obtained. Having the data of density, porosity, packed bed dimensions, and flow rates versus pressure settings used, the specific surface area by the gas permeability method can be calculated. Determining cement fineness that way is an alternative to ASTM C204 method and no Blaine or similar apparatus is necessary. An improved theoretical approach has been proposed for packed beds and rock cores permeability. Additional capabilities and specialized pycnometers are continuously being developed and more detailed information can be found in applications notes and materials presented on our websites.

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