InstruQuest Inc.

Providing Complete Solutions

Technical Note
March 2018

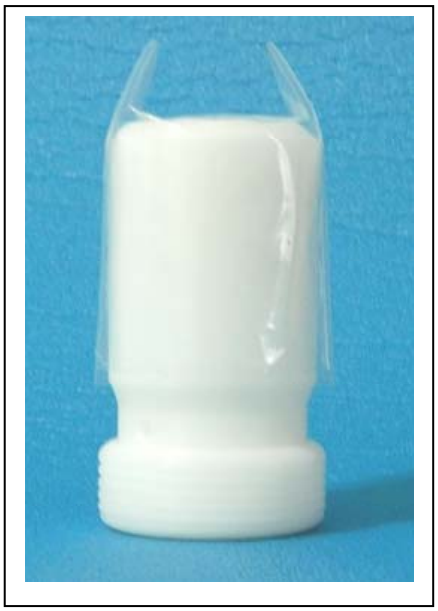
By Jan Malczyk and Teresa Malczyk

The solution for handling "difficult" samples when measuring true volume (density) using gas (helium) pycnometers

There is a large spectrum of samples ranging from solids to almost liquids that present practical problems in measuring their density. They often do not conform to typical classification as solids where the gas (helium) pycnometers are used and they cannot be handled by density meters that are designated for freely flowing liquids. Moreover, when such samples are placed into a sample holder, they present a practical problem of sample holder contamination and subsequent cleaning of such sample holder, often having to use aggressive solvents and facing all safety and environmental issues.

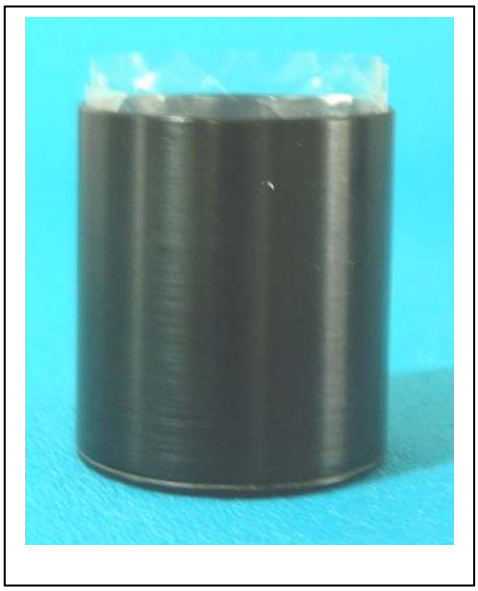
A simple solution for handling such samples is to use disposable inner liners and that is easy to implement in gas pycnometers made by InstruQuest thanks to special designs of sample holders. The sturdy wall of the sample holder (1.3mm thickness) allows for usage of machined cover with thin O-ring on its side to ensure hermetic contact between the cover and the holder wall. The top of the cover is equipped with a fritted metal filter that can be of different porosity rating, e.g. 2, 5, 10, 20, or 40 microns. All metal components of the sample holder are chemically treated to ensure corrosion resistance surfaces.

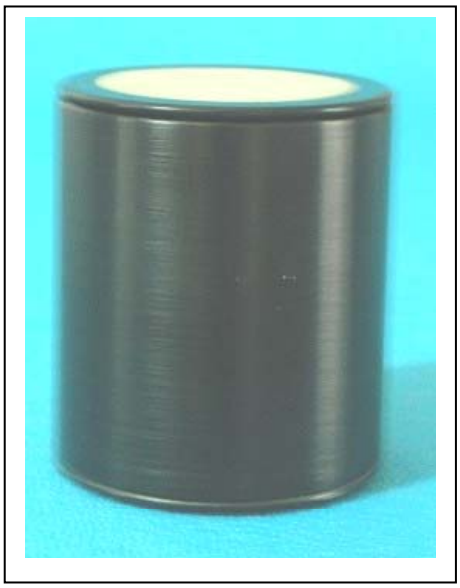
Easily commercially available polyethylene bags offer good chemical resistance and can be acquired with different wall thicknesses at very low cost. They can be easily formed into tubular shape using a specially designed tool presented on photo below (left hand side).



The edges of the bag can be folded to the center and the bag with the tool can be easily inserted into the sample holder (see photo above on the right hand side). The design of the tool allows for its easy removal and leaving the formed bag inside the sample holder (see the photo below on the left hand side). The

excess of the bag edges can be folded to the inside and the cover installed onto the sample holder. The photos below on the right hand side illustrate the results of the described actions.





Now the sample holder with the inner lining needs be inserted into the sample chamber to measure the volume of the sample chamber without sample. After the completion of this part of experiment (paused when wait tag = 0 is encountered), the user needs to remove the sample holder from the sample chamber, weigh it, and the mass of the sample holder recorded. Next, the cover need to be removed and the edges of the bag should spread outside to allow for convenient addition of sample. Once the sample is added, the edges can be folded back to the inside again and the cover installed. The sample holder with the sample needs to be re-weighed and the sample mass determined. Once the next part of experiment with the sample is completed, the volume of the sample is determined by the pycnometer. The sample mass can be entered into the software any time before the report is printed.

Only the designated sample holder cover with the O-ring should be used as it will not allow lifting up the cover by the squeezed liner. More importantly, the fine filter will work in such “air-tight” construction as a buffer reducing any pressure shocks occurring during the experiments. There are however additional aspects of working with plastics and semi-solid samples in general. Any sample that is easily compressible or exhibits net sorption/desorption hysteresis during pressurization/depressurization cycle may require a longer equilibration time to achieve better stabilization of pressure changes versus time. That is relatively easy to accomplish by viewing the equilibration profiles in the software and modifying the length of programmable equilibration time. For easily compressible sample, a lower pressure can be used although it has adverse effect on accuracy in general. A well researched method needs to be applied to measurements of true volume (density) by a gas pycnometer of a given class of samples. If no other simple technique can be used for determination of density of semi-solids samples, the presented above approach provides an inexpensive and convenient solution of handling “difficult” samples, like pastes, slurries, gels, etc.