

Sample holders for true density measurements of ultra-fine materials by HumiPyc gas (helium) pycnometers

Several new types of sample holders of proprietary design have been developed by InstruQuest Inc. for measurements of true volume (density) of very fine powders, in the range of nano-micro particles sizes. The gas (helium) pycnometers operating on the principle of gas expansion require steps of pressurization, depressurization, and venting to atmosphere in a single measurement cycle. Such cycles are often repeated many times for statistical purposes. Consequently, the disturbance caused by the pressure swings creates a problem with sample elutriation, especially with fumed silica or similar materials of submicron particle sizes. In addition to the general-purpose sample holders used in HumiPyc pycnometers, that have pressed-in cover with metal fritted filter installed, new types of sample holders are being offered.

1. Large sample holders with fixed porosity filters

The improved design features addition of a thin O-ring to the sample holder cover to ensure air-tight closure between the cover and the holder barrel. The cover can have installed one of several choices of fine metal filters of different porosities: 2, 5, 10, 20, or 40 microns. The SS metal filter has diameter of 38 mm and 1.6mm thickness. The large diameter allows for uniform gas flow in and out and dampens quick pressure changes occurring externally to the sample holder. In addition to the usage of proportional valves in HumiPyc pycnometers for gradual changes, this sample holder, especially with 2-micron SS filter, ensures a high degree of protection against sample removal during measurements.

The picture below shows an example of such filter with parts numbers listed for different filter micron size. For protection against some reactive materials, the aluminum filter is black anodized, but for some special applications it can be also supplied without anodizing.



Large Sample Holder, Anodized, Fixed Porosity of 2, 5, 10, 20, or 40 microns:

P/N: LSHAFP2M – 2 microns
P/N: LSHAFP5M – 5 microns
P/N: LSHAFP10M – 10 microns
P/N: LSHAFP20M – 20 microns
P/N: LSHAFP40M – 40 microns

2. Large sample holders with variable porosities filters (User-defined filters)

Development of special materials often necessitates individual approach to filter design. Multi components filters allow the user to construct a filter of desired porosity that best addresses a given application. The cover usually consists of two parts, the bottom, and the top nut. The bottom has the O-ring for airtight connection with the holder barrel. The user-prepared filter structure is placed onto the flat part inside the

nut, which subsequently is screwed into the bottom part. The nut and the bottom part are easy to insert and remove from the main body of the sample holder. It can be reused and the filter construction modified at any time by the user.

The filter structure can vary, but typically a filter (or filters) of specific porosity are placed between metal (SS) mesh discs for mechanical supports and pressed inside the nut. To allow for large selection of filters, the standard size of 47 mm round filters can be utilized and such materials and they are available from various vendors worldwide. Many filter sizes in microns, like 0.1, 0.45, 1, 2, 8, and others from various materials can be used. The picture below shows example of such sample holder design. Since it is up to the user to use a particular filter sizes, the SS mesh circles and some examples of fine filters, like 1 or 8 micron are usually provided.



Large Sample Holder, Anodized, Variable (User-defined) Porosity

P/N: LSHAVP

Standard filter(s) of 47 mm Diameter of various porosities can be used, e.g. 0.1, 0.45, 1, 2, etc.

Other particular versions of the filter design also feature one of the SS fritted filters installed permanently in the cover, or a second one in the mating bottom part. Again, in between the surfaces, the user defined filter or their combination can be placed for additional filtering capabilities. An example of such sample holder with 2 micron SS filter installed in the nut is shown in the picture below.



Large Sample Holder, Anodized, Variable (User-defined) Porosity with Fixed Porosity SS filter

P/N: LSHAVP2M – 2 microns

P/N: LSHAVP5M – 5 microns

P/N: LSHAVP10M – 10 microns

P/N: LSHAVP20M – 20 microns

P/N: LSHAVP40M – 40 microns

Standard filter(s) of 47 mm Diameter of various porosities can be used, e.g. 0.1, 0.45, 1, 2, etc.

In the purpose of restricting even the tiniest particles to flow out of the sample holder, one should keep in mind that such filtering barriers also restrict gas flow in and out of the container. It is not expected to focus on speed of experiment as the equilibration profiles for establishing a stable value of gas pressure in the sample chamber will most likely take longer with a particular filter construction than it would be without it. Since the equilibration profiles versus time can be observed in real time, the most suitable time interval can be selected. It should be noted, that usage of the 24-bit data acquisition in the HumiPyc pycnometers allows to observe much smaller pressure changes than in other pycnometers that use much smaller resolution, and therefore, the time can be usually longer if stricter equilibration criteria are declared by the user.