

APPLICATION NOTE

MEASURING HOLLOW FIBRE SAMPLES WITH POROLUX™ POROMETERS

The characterisation of hollow fibre membranes by gas liquid porometry is fraught with experimental challenges. The fibres are often delicate, and subject to stretching, deformation, and even rupture.

The POROLUX™ 1000 addresses these challenges with a specially designed Sample Holder for Hollow Fibres (SHHF) (Figure 1) that permits easy attachment of hollow fibres in one of two ways - depending on the direction of the gas flow - either from inside to outside or from outside to inside. The design allows for easy adaptation and testing of fibres of various internal and external diameter.



Figure 1: overview of the SHHF

POROMETRY MEASUREMENTS OF HOLLOW FIBRES: OUTSIDE-IN

The end of a fibre must be sealed with glue (e.g. epoxy, acrylate), such that test gas enters the fibre from one end and can only flow out through the walls via the lumen. This is the outside-in test approach. The open end of the fibre is sealed inside the bore of the bottom connector, with the threaded part facing UP so that the fibre projects ABOVE the threads (see figure 2). Glue is then used to avoid gas escaping through the gap between the fibre and the bottom connector. It is recommended to let the glue dry overnight before wetting the sample prior porometry measurements. The time required for the glue to dry depends on its composition (we advise to follow the instructions of the glue manufacturer).



Figure 3: SHHF attached to the POROLUX™ 1000 for outside-in measurements.

After the glue has dried, the next step is wetting of the sample by immersion in the selected wetting liquid. Subsequently, the bottom part of the sample holder is screwed into the top part of the sample holder, so the fibre remains INSIDE the sample holder. The entire unit is snapped on to the POROLUX™ and after setting the optimal measurement parameters, the test can proceed. Once attached to the POROLUX™ the sample holder looks as shown in figure 3.

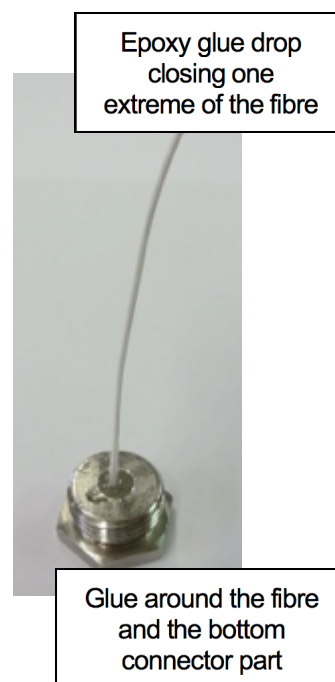
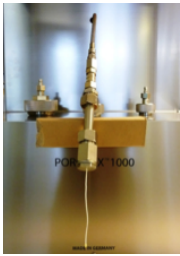


Figure 2: detail of the gluing of the fibre for outside-in measurements

POROMETRY MEASUREMENTS OF HOLLOW FIBRES: INSIDE-OUT

As before, one end of the fibre (bottom) must be sealed with glue such that gas may only exit vial the fibre walls. On the top part of the fibre, glue must be added around the sample holder to attach the fibre, to the part, while leaving the inlet open (no glue may block gas entry to the fibre). This must be done such that there are no gaps between the sample holder and the fibre, so the gas does not escape on the sides (see figure 4). Again, it is advised to let the glue dry overnight before wetting the sample prior measuring.



Figures 4 and 5: detail of gluing of fibre for inside-out measurements

The top part of the sample holder is then attached to the bottom with the threaded part facing UP and the fibre REMAINING hanging BELOW, OUTSIDE the sample holder. Once attached to the POROLUX™ the sample holder looks as shown in figure 6.

Regardless of the direction of the flow, it is sometimes necessary to glue multiple fibres together in order to achieve a measurable flow rate (figure 7). In these cases, the obtained pore size distribution will be an average of the pore size distribution of the individual fibres.



Figure 7: multiple fibres glued together.

EXAMPLE: POLYMERIC HOLLOW FIBRE COMMERCIAL MEMBRANES

GLP measurements of commercial polymeric MF hollow fibre membranes were carried out by using a POROLUX™ 100, based on the pressure scan method (the increasing pressure and the resulting gas flow are measured continuously during a test). The maximum operating pressure of the device is 7 bar (100 psi), which permits measuring pore sizes between 0.091 µm and 500 µm. The wetting liquid used was Porefil and the samples were measured up to a final pressure of 1.8 bar at a speed of pressure increase of at a pressure speed of 3.3 mbar/s. The direction of the flow was inside-out and the first bubble point (FBP) was calculated at the pressure corresponding to the first measurable flow. The resulting wet and dry curves for three replicates (three measurements of three different fibre pieces of the same sample) are shown below. From these curves it is possible to obtain information on the First Bubble Point, the Mean Flow Pore (MFP) and the Smallest Pore (SP) sizes.

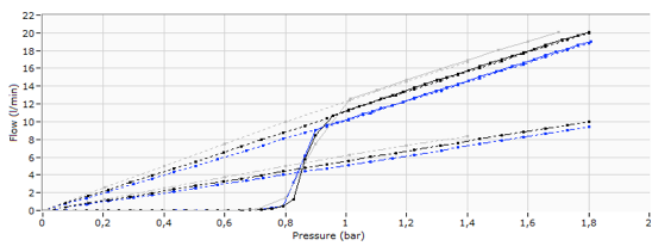


Figure 8: wet, dry and half dry curves of inside-out measurements of polymeric hollow fibre membranes with the POROLUX™ 1000

	First Bubble Point	Mean Flow Pore	Smallest Pore
Average (µm)	0.78	0.536	0.48
SD (µm)	0.02	0.007	0.02

Table 1: FBP, MFP and SP of inside-out measurements of polymeric hollow fibre membranes with the POROLUX™ 1000

The gas flow through the sample is used to calculate the flow distribution with respect to the flow, as shown below.

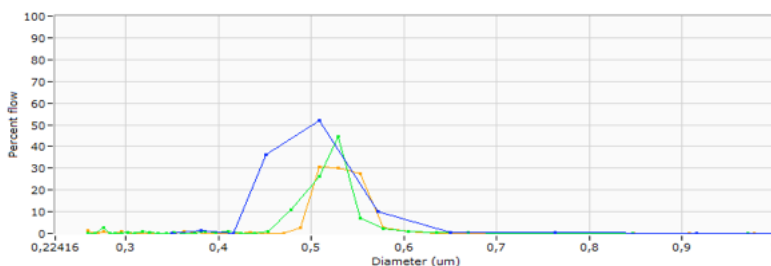


Figure 9: flow distribution curve of inside-out measurements of polymeric hollow fibre membranes with the POROLUX™ 1000