

Theoretical considerations and methodology

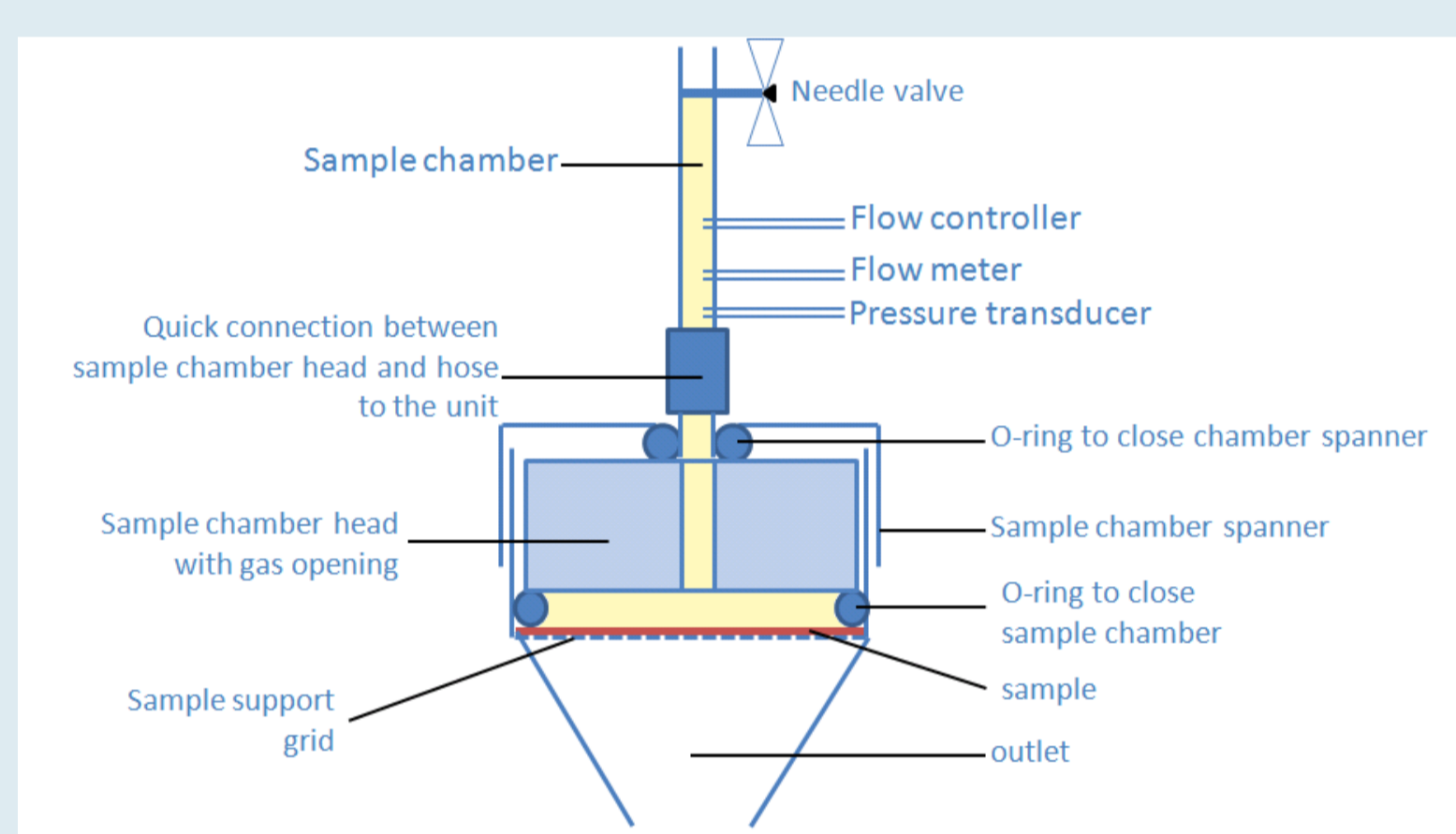
With porometry through pores inside a sample are characterized. Through pores are open on both sides of a filter or a membrane and therefore determine the functionality. Important parameters are the largest pores, the mean flow pore size and smallest pores. The smallest pores determine the diameter of the smallest particles that will pass through the filter, the mean flow pore size or **MFP** represent average pore size. The largest pores, historically called the first bubble point or **FBP**, determine the size of the largest through pores in the filter. Besides these characteristics also the pore size distribution – showing the spread of the different pore diameters – and the gas permeability can be calculated.

Already in 1921, Washburn considered the existing relationship between pressure and pore size, whether a liquid is pushed into the pores as in mercury porosimetry or a liquid is pushed out of the pores as is the case in porometry. The following relationship is valid for these methods:

$$\text{Pressure} = 4 * \gamma * \cos \theta * (\text{shape factor}) / \text{diameter}$$

With the pressure in bar, the diameter in μm , γ the surface tension of the wetting liquid, θ the contact angle of the liquid on the solid. The shape factor is a parameter depending on the shape and the path of the pore inside the material.

Capillary flow porometry uses a liquid that spontaneously flows through pores of a sample. The sample is then placed inside the instrument and the pressure is gradually increased. At a certain point the liquid is pushed out of the material and the resulting pore size diameter is then calculated using the above equation. A typical setup is shown in the picture below.

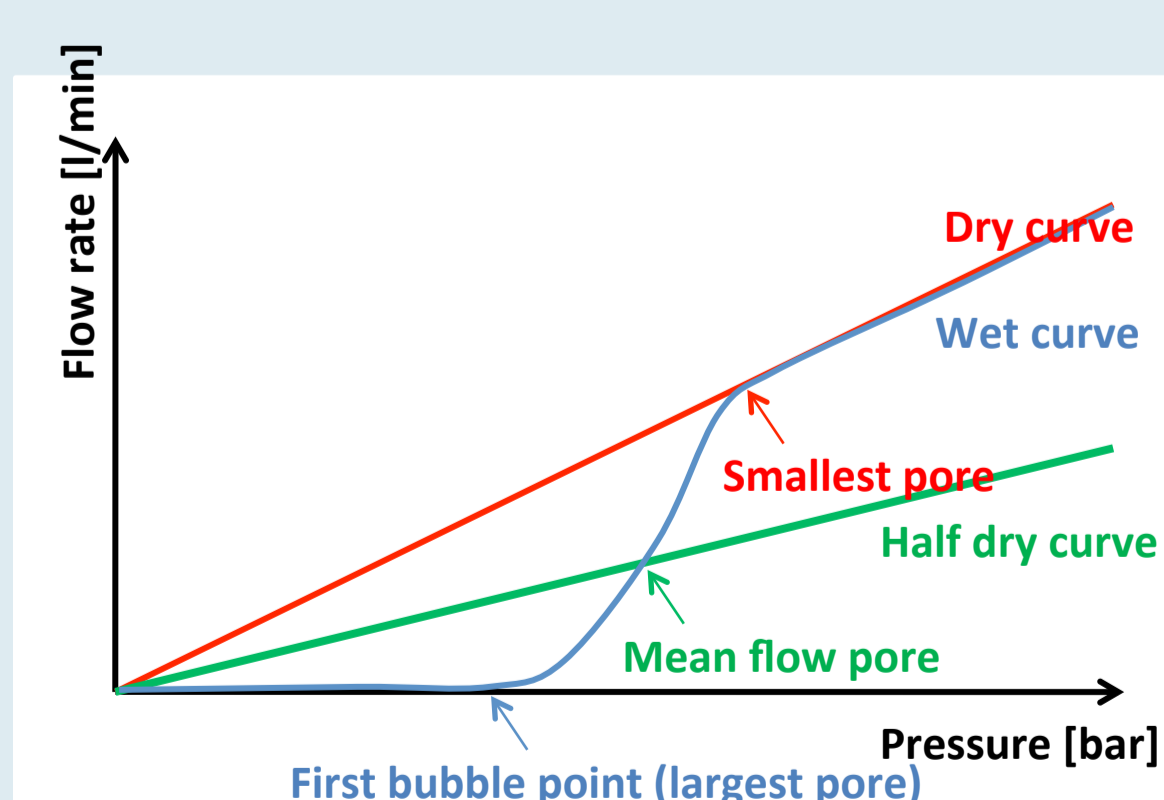


The surface tension γ is a measurable physical property and is available for many liquids. The contact angle θ however depends on the interaction between the material and the wetting liquid. Typical wetting fluids used in porometry are perfluoroethers. They have a low surface tension and a contact angle of 0° with nearly all materials.

Wetting liquid	Chemical composition	Surface tension dyn/cm*
Porefil	Perfluoroether	16
Galpore	Perfluoroether	16
Silpore	Silicone oil	20
Water		~72
Methanol		22.50

* Measured at 20°C in air

A standard analysis consists of the measurement of two curves: a wet curve measured after impregnation the sample in the wetting liquid and the dry curve measured on the same not impregnated filter. The pressure is increased in the desired pressure range. A full porometry measurement typically generates the following graph from which all pore characteristics are calculated.



Our porometer technology

Porometer.com offers two different types of porometers. The Porolux 100 series uses a **pressure scan** method. In this method a single valve is opened during a measurement and the pressure and the resulting gas flow are measured continuously. This method is fast and typically generates very reproducible results. Therefore the Porolux 100 series is very suited for QC type of work. A drawback is that samples with complex pore paths are difficult to measure. Due to the method these porometers are less accurate at higher pressures. A special version of the Porolux 100 is available for materials with large pores such as of textiles and non-wovens.

	Porolux 100	Porolux 100NW	Porolux 200
Measuring principle	Pressure Scan	Pressure Scan	Pressure Scan
Max pressure	7 bar	4 bar	14 bar
Min pore ⁽¹⁾	91 nm	160 nm	45 nm
Max pore ⁽²⁾	300 μm	500 μm	300 μm
Max flow	100 l/min	200 l/min	100 l/min
Sample holder	25 mm	25 mm	25 mm
Pressure sensors	8 bar	5 bar	15 bar
Flow sensors	5-100 l/min	10-200 l/min	5-100 l/min

⁽¹⁾ Using Porefil
⁽²⁾ Using silicone oil

The Porolux 1000 series uses a different, **pressure step** method to measure pore diameters. The inlet valve for the gas is a large, specially designed needle valve that is opened with very accurate and precise movements. To increase pressure, the valve opens to a precise point and then stops its movement. The pressure and flow sensors will only take a datapoint when the used defined stability algorithms are met for both pressure and flow. In this way, the Porolux 1000 detects the opening of a pore at a certain pressure and waits until all pores of the same diameter are completely opened before accepting a datapoint. This results in very accurate measurement of pore sizes and allows a calculation of the real pore size distribution. The Porolux 1000 comes with pressure transducers up to 35 bar, allowing minimum pore size measurements down to 18 nm ⁽¹⁾. This series also come with more options such as multiple bubble point detection algorithms, liquid permeability, hydrohead and special sample holders for hollow fibers, large samples, etc.

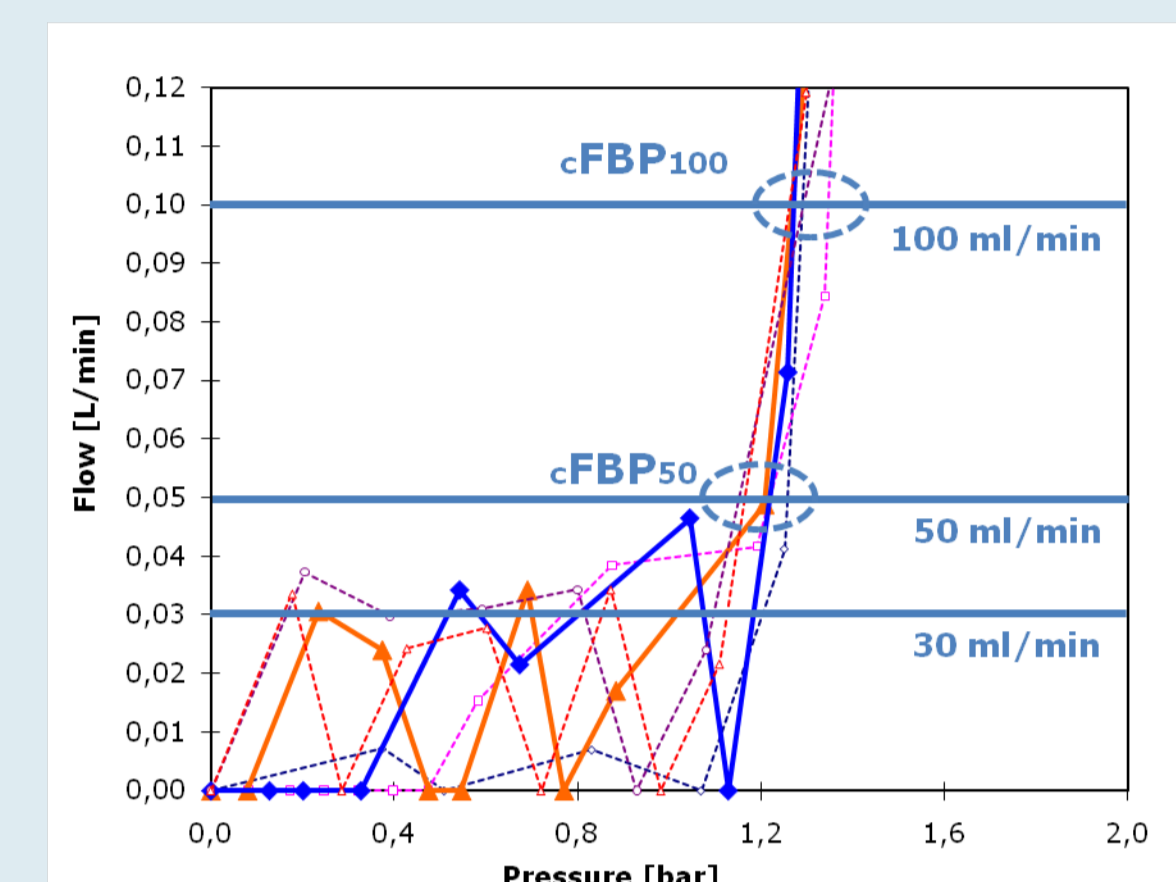
	Porolux 1000LP	Porolux 1000
Measuring principle	Pressure Step	Pressure Step
Max pressure	8 bar	35 bar
Min pore ⁽¹⁾	80 nm	18 nm
Max pore ⁽²⁾	500 μm	500 μm
Max flow	100 l/min	200 l/min
Sample holders	25 mm	13-25-47 mm
Pressure sensors	1-10 bar	2-50 bar
Flow sensors	5-100 l/min	10-200 l/min
FBP regulator	5-30 ml/min	5-30 ml/min
Measured FBP	Yes	Yes
Calculated FBP	Yes	Yes
Liquid permeability	Option	Option
Hydrohead	Option	Option
Comfort kit	Option	Option
Large sample sample holder	Option	Option
Hollow fibers sample holder	Option	Option

⁽¹⁾ Using Porefil
⁽²⁾ Using silicone oil

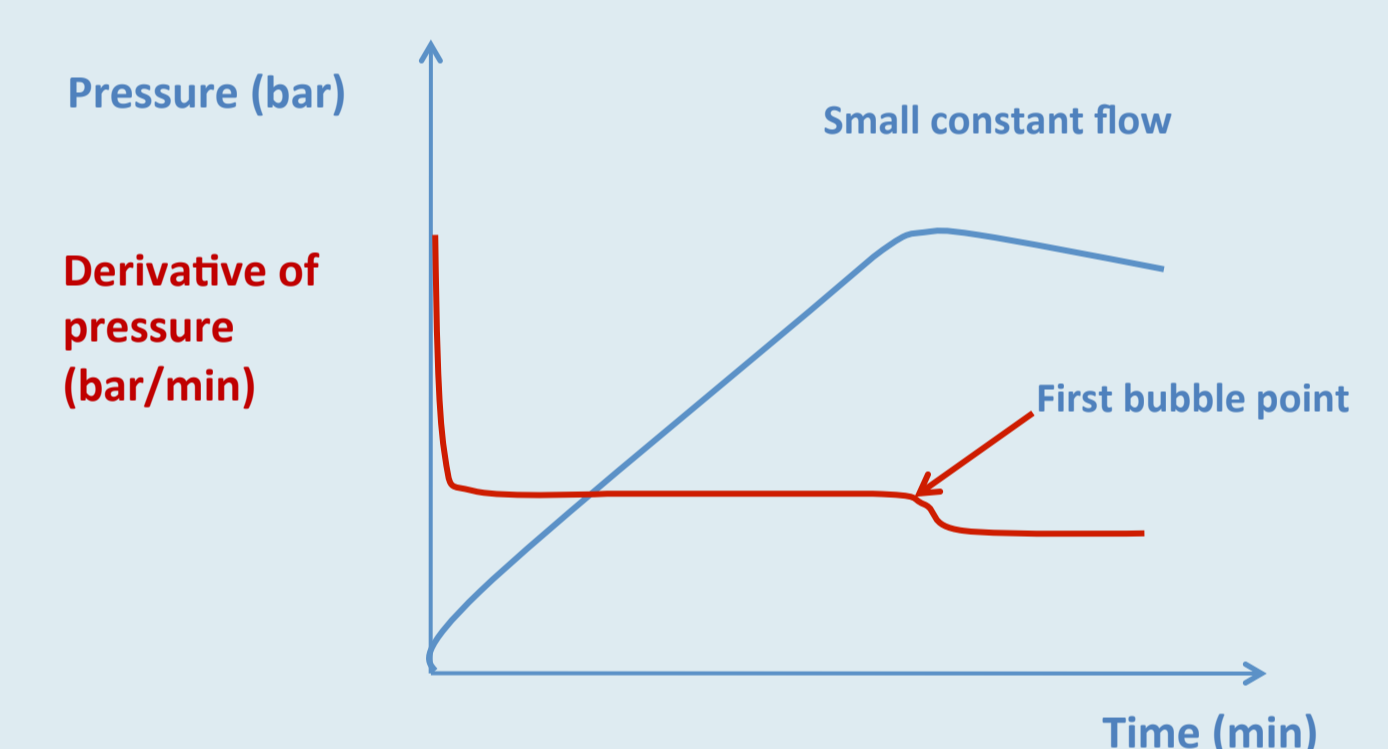
All Porolux models are built on the latest, universally available, PLC technology and run on LabVIEW software. All instruments use network connection facilitating diagnosis and service via the internet.

Bubble point measurements

One of the most important parameters measured by a porometer is the first bubble point or FBP. This point defines the largest pores present inside the material. ASTM F-316 defines the FBP as the pressure at which the first continuous bubbles are detected. With a pressure scan porometer the FBP can be defined at different flows, e.g. at 30, 50, 100 ml/min. Because with this approach there is already flow at the FBP, by definition, this calculated FBP is always smaller than the real bubble point and thus the **calculated FBP** never represents the real opening of the largest pores.



There is another, **measured bubble point** approach for detecting the largest pore. In the Porolux the fully wetted sample and sample chamber all the way up to the needle valve form a closed system. Therefore, if we increase the pressure on the sample using a small, constant flow of gas towards the sample chamber, the pressure will slowly increase. As the volume is fixed, this constant flow will result in a linear rise of the pressure above the sample. At the moment the first and largest pore is opened, there will be a change in the linear pressure increase. This change can be regarded as the true first bubble point of the material. This method to measure the FBP shows an excellent reproducibility.



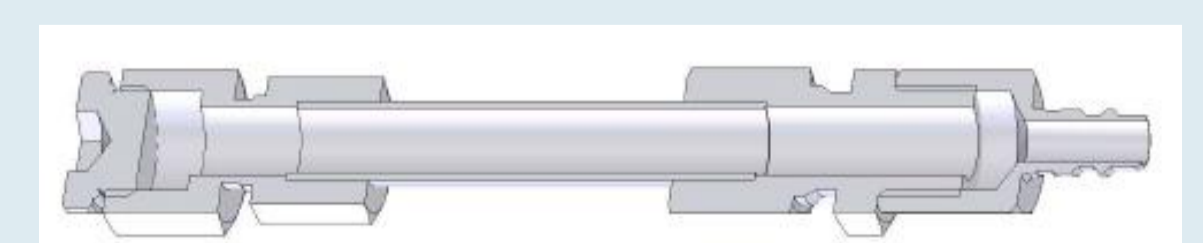
Accessories

Most of the accessories are only available for the Porolux 1000 series because for many of these accessories high pressures and stabilization of pressure and flow are highly desirable.

Besides gas permeability, users can also choose for **liquid permeability**. This option allows to measure the permeability of non-corrosive liquids through a sample.

For measuring the impermeability of a material and a liquid, there is the **hydrohead** option. This option simulates the well-known test measuring the height of a water column at which the sample cannot withstand the water pressure any longer.

The standard **sample holder** are 13, 25 and 47 mm, but all other size sheet sample holders are available. We also design and make sample holder for special applications and special sizes. We have developed specific sample holders for research on **hollow fiber** materials. Because of their layered structure, hollow fibers need to be measured from the inside towards the outside and the other way round. Our special hollow fiber sample holders allows the sample to be measured both ways. The sample holder is safe at high pressures, is easy to use and to clean and can be used multiple times.



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