The POROLUX™ “true” First Bubble Point detection mode

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The POROLUX™ 1000 is a research grade capillary flow porometer with very pressure and flow control and accuracy. In the POROLUX™ 1000 a porous sample is inserted, wetted with a so-called wetting liquid. The sample is usually a flat round disk, three different diameters are standard: 13, 25 and 47 mm. Custom made sample holders are optional. The wetting liquid is a liquid which fills up all pores of the sample. Good contact between the liquid and the sample is therefore necessary. Different liquids can be used, the surface tension should however be exactly known and entered in the software prior to the measurement. This value is used for the calculation from pressure to pore size.

A typical capillary flow porometer gradually builds up a nitrogen or other inert gas pressure in between two chosen boundaries. Flow meters follow the flow of gas through the sample. In the graphs, gas flow is shown as a function of pressure. The wetting liquid is pushed out of the large pores at lower pressures, small pores require a higher pressure in order to be emptied. First such a wet curve is measured, after which the same experiment is performed for a complete dry sample.

The “true” first bubble point

Bubble points were for a long time measured in capillary flow porometers by the method described in ASTM F-316 as the pressure at which predefined flow was measured. The POROLUX™ 1000 can be operated in a calculated of in a measure bubble point mode. In the calculated bubble point mode, the POROLUX™ 1000 offers several predefined flow to calculate the first bubble point. In the “true” first bubble point bubble point detection mode, the POROLUX™ 1000 meets the original, physical definition of the bubble point.

The first developed bubble point testers measured a bubble point in the more exact way: a filter was placed in a small tank filled with water or another solvent and connected to a pressure medium allowing a gradual build up of the pressure (see figure 1). The pressure at which the first continuous stream of gas bubbles is observed, is the bubble point pressure. This pressure can be easily calculated into the bubble point pore size.

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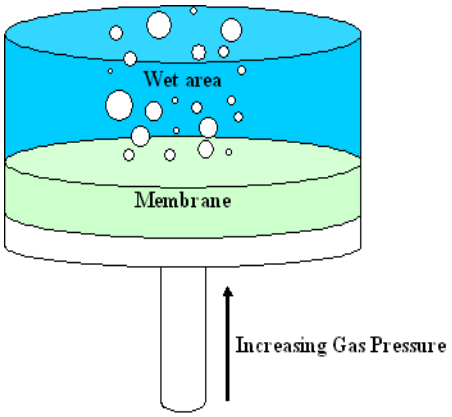


Figure 1: Scheme of a bubble point tester

Inside the POROLUX™ 1000, there is - besides pressure and flow sensors - a small flow controller. Until the first pore is opened, the whole sample compartment forms a closed system (see figure 2), so a small continuous flow will result in a small linear pressure increase.

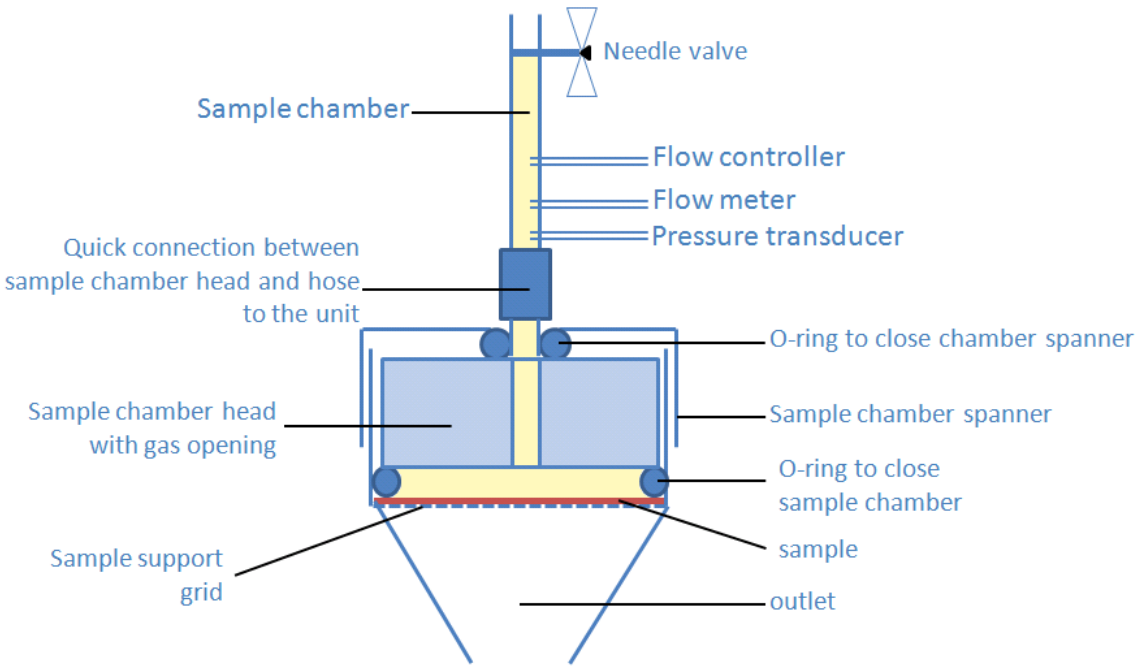


Figure 2: Schematics of the interior of the sample chamber of the POROLUX™ 1000

In the “true” first bubble point detection method, the user can select a flow between 5 and 30 cc/min. As a result of this gas flow, there is a linear pressure increase in the sample chamber. When the first (largest) pore will be opened, a sudden decrease in the linear pressure increase will be observed. Therefore the first derivative of pressure to time is continuously monitored. The “true” first bubble point pressure is the pressure at which the first derivative starts to deviate from a straight line. The magnitude of this deviation is user selectable as a percentage deviation from a straight line (see figure 3). The corresponding pore size to this pressure is then defined as the “true” first bubble point.

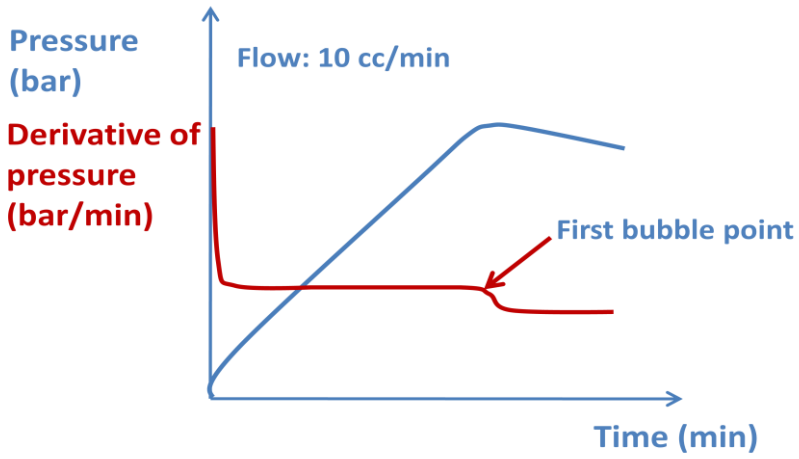


Figure 3: Linear pressure increase and first derivative of pressure as a function of time for a typical bubble point test.

In table 1, the results of the “true”, measured first bubble point measured on two different samples with several repeated runs is shown. Identical bubble point settings were used: 30 ml/min for the gas flow towards the sample and a requested deviation on the pressure signal of 50%. As can be seen, this method generates very reproducible results.

	Measured FBP sample 1 (µm)	Measured FBP sample 2 (µm)
Test 1	1.57	1.55
Test 2	1.56	1.61
Test 3	1.56	1.56
Test 4	1.57	1.57

Table 1: Measured bubble points using the bubble point method of the POROLUX™ 1000.

The POROLUX™ 1000 can also be operated in a “bubble point only” mode, where the instrument only measures the fist bubble point. In the “Full porometry” mode, there is the “Skip bubble point” option. When this button is NOT highlighted, the POROLUX™ 1000 operates in the “true” first bubble point mode as described above. The first recorded datapoint will be the measured, “true” first bubble point (see FBP in figure 4). Once the first bubble point has been found, the POROLUX™ 1000 will increase the pressure from the FBP to the end-pressure in the number of interval requested by the user to obtain the wet and dry curves. A datapoint will only be taken if the required stability for pressure and flow are reached.

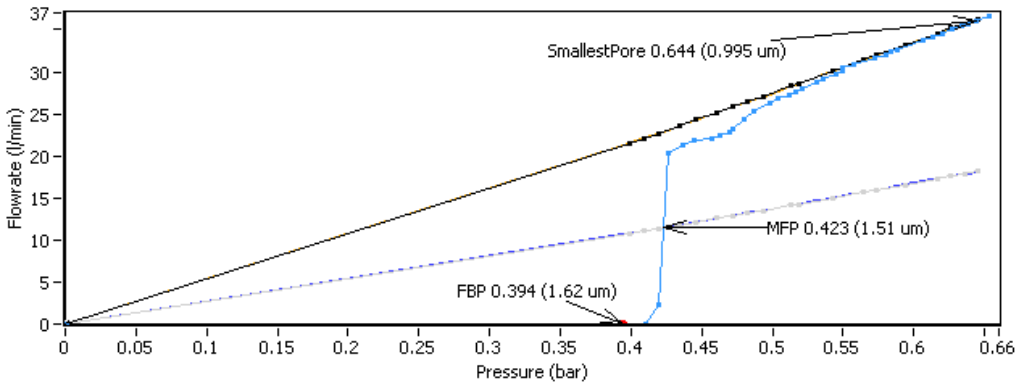


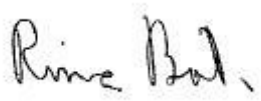
Figure 4: Measured “true” first bubble point (FBP)

If the “Skip first bubble point” is highlighted, the POROLUX™ 1000 will operate in the “calculated” first bubble point mode. In this mode the POROLUX™ 1000 operates in the ASTM F 136 method where the first bubble point will be calculated from the pressure at which a defined flow is measured. Calculated first bubble points at different flows (30, 50 or 100 ml/min) or by differential size accounting (which corresponds to the start of the largest increase in flow for a filter) are suggested.

A sample measured with a “calculated bubble point” instrument will always show a higher bubble point than the same sample analyzed with a measured, “true” bubble point” instrument (in the first approach, there is already e.g. 50 ml/min flow, thus the pressure is already higher, consequently the diameter will be smaller).

Conclusion

The POROLUX™ 1000 offers multiple modes to find the first bubble point. With the measured bubble point method, the POROLUX™ 1000 comes close to the original, physical definition of the bubble point, namely the first pressure where a pore is opened. The sensitivity and the speed for measuring the first bubble point detection can be changed by the user. This allows the POROLUX™ 1000 to find the “true” first bubble point in a wide range of materials.



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