The POROLUX™ "calculated" First Bubble Point detection mode

By dr. Bart Rimez

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.

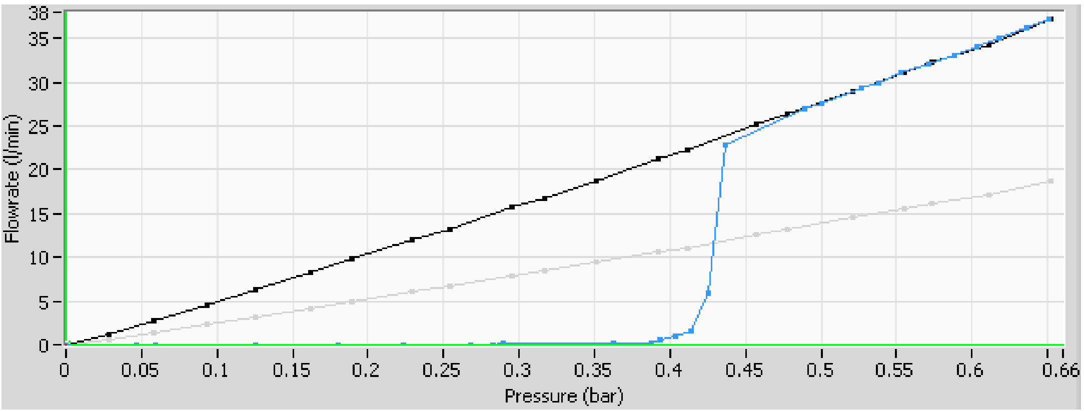
Bubble point detection

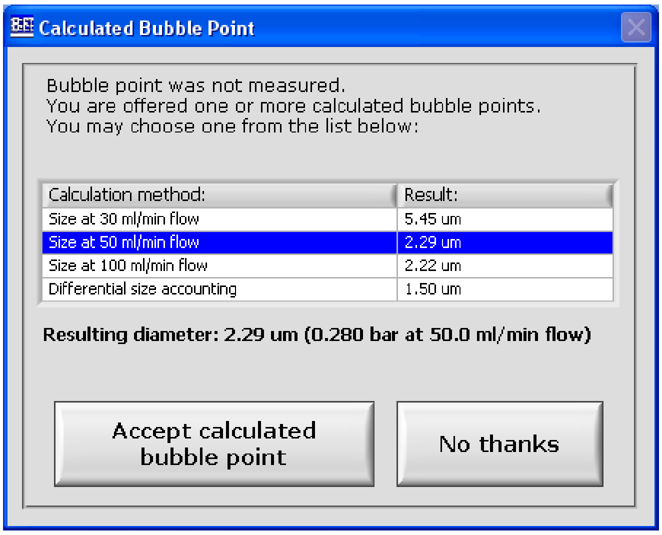
The bubble point is defined as the pressure required to blow the first continuous bubbles detectable through a sample. This bubble point test can be performed by pre-wetting a sample, increasing the pressure of gas above the sample at a predetermined rate and watching for gas bubbles downstream to indicate the passage of gas through the maximum diameter pores in the sample (ASTM F316). The first detected gas flow is defined as the bubble point. Home build devices still use this visual detection of the first (stream) of bubbles; the pressure at which this first (stream) of bubbles happens is used to calculate the bubble point. Automated methods use flow meters detecting a certain minimum flow. The first Coulter porometers defined the bubble point as the pressure at which 100 ml/min was measured. In the mean time, mass flow meters have become more sensitive and thus lower flows can be set (e.g. 30 or 50 ml/min).

This implies that there is no uniform definition of bubble point, one should always specify the minimum flow that was used to calculate the bubble point (there are also mathematical methods searching the onset of the flow increase in the flow/pressure graph).

A drawback of this approach is that there has to be a minimum flow in order to detect the bubble point. As a result, the bubble point is calculated at a pressure that is already slightly too high, and therefore this calculated bubble point will be too small. To overcome this drawback, the POROLUX™ 1000 can also be used in a "true" measured bubble point method (see below).

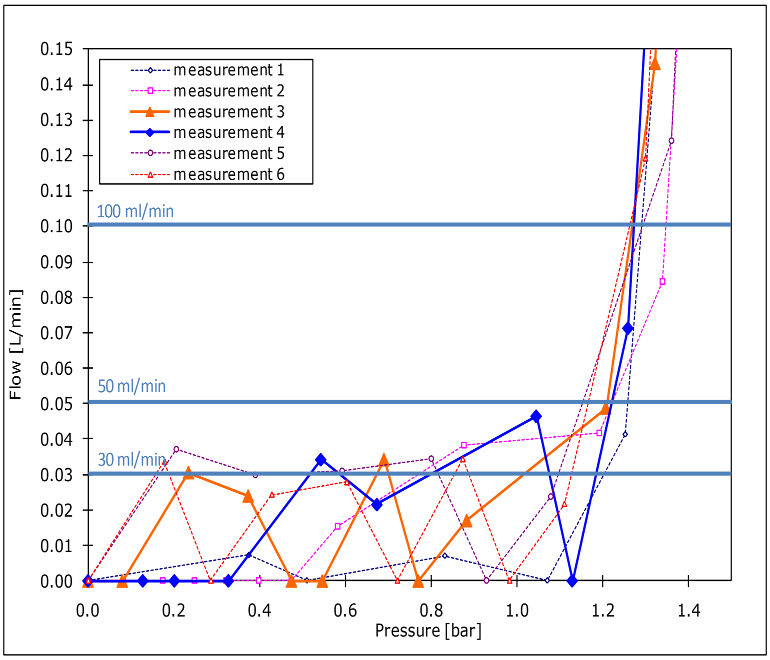
Another drawback of this method is often that a real datapoint is seldom measured at the exact chosen value of 30, 50 or 100 ml/min. Therefore, these bubble points are usually extrapolated or the datapoint nearest to the selected bubble point flow is taken. The data below shows an example of a measurement with gradually increasing pressure. After the measurement of the wet curve (blue) and the dry curve (black), a window popps up offering several options for the calculated bubble point (see window):





Obviously in this calculated bubble point method, the lower the minimum flow selected, the closer we are to the real bubble point, 30 ml/min will be closer to the true bubble point than 100 ml/min.

The influence of the selection of this minimum flow is shown in the graph below. The experiments show a test on 6 different samples of an identical material having excellent reproducibility for the wet curves.



Using the predefined minimum flow method to calculated the bubble point, the first datapoint where the flow though the sample crosses the predefined minimum flow is determined as the calculated bubble point. One should know however that in a porometer, both pressure and flow sensors are physically positioned above the sample. Pressure is built up by adding more gas to sample chamber. Adding more gas implies a certain gas flow which will be detected by the gas flow meter (because it is positioned above the sample).

So, it is possible to detect a small gas flow before the first gas is blown through the pores of the sample. Depending on the pressure and gas flow stabilization time (predefined by the user), the flow can exceed easily 30 ml/min gas flow, without actually having opened the largest pore. This is exactly what happened in these experiments: the flow exceeds 30 ml/min, but the calculated bubble point values at 30 ml/min have absolutely no physical meaning and large errors occur when the bubble point is calculated with this minimum flow. At flows of 50 and 100 ml/min, some pores are really opened and give a more stable (and more reliable) calculated bubble point. The table below gives an overview of the bubble point calculated with these different pre-set flows:

	Bubble point at 30 ml/min [µm]	Bubble point at 50 ml/min [µm]	Bubble point at 100 ml/min [µm]
Measurement 1	0.38	0.37	0.36
Measurement 2	0.60	0.38	0.34
Measurement 3	2.00	0.38	0.36
Measurement 4	0.89	0.38	0.36
Measurement 5	2.86	0.40	0.36
Measurement 6	2.69	0.39	0.36
average	1.57	0.38	0.36
standard deviation	1.09	0.01	0.01

However, with this method, you do not actually determine the “true” first bubble point. As mentioned above, this calculated bubble point is found at a pressure that is already slightly too high (there is already flow), and therefore this calculated bubble point will be smaller than the “true” bubble point.

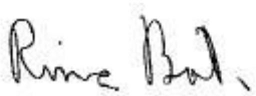
To measure the “true” bubble point we need to use another operating mode where a special circuit is used to apply a low, constant flow rate (user selectable between 5 and 30 cc/min). Because we are in a pre-bubble point phase, all pores are blocked by the wetting liquid, thus the constant flow results in a linear increase of the pressure (think about blowing up a balloon at a constant blowing rate). At a certain pressure, the largest pore will be opened and a sudden change in the linear pressure build-up will be detected. This “true” bubble point detection is described in another application note.

Conclusion

There is no uniform definition of bubble point, therefore it should always be mentioned how the bubble point was determined. The POROLUX™ 1000 offers several modes to determine the bubble point.

With the indirect way, the user can choose different flowrates to be used as “first flow” for the definition of the bubble point.

The POROLUX™ also has the ability to measure the “true” bubble point with a more theoretical approach where the actual first opening of a sample is measured. This can be measured with several sensitivities in order to detect this point as exact as possible.



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