

## Oil and filtration

# Questions on filter pore size analysis

**T**he costs and delays experienced in extracting oil can be increased by mistakenly using a filter with the wrong sized pores. Graham Rideal, of Whitehouse Scientific tells us more.

In the petrochemical industry, crude oil extraction is a very expensive business in that the rigs cost approximately \$250,000 a day to hire. On top of this is the cost of sinking stainless steel pipes up to 10,000 metres to the oil seam. A critical part of the extraction is the sand screen at the end of the pipeline. If the pore size is too large, sand contamination can damage both the pumps and the extraction pipes. If the pore size is too small, the filter can plug. Either of these two scenarios can 'junk' an oil well at a cost in excess of \$20 million, so accurate measurements of pore sizes are of paramount importance.

### Scientific Measurements

In any scientific measurement three very important questions must be asked in order to place any confidence in the results. Firstly, is the technique accurate, secondly is it repeatable and thirdly, is it traceable to an international standard.

In the case of pore size measurement, the two most popular methods are Porometry and challenge testing. In the former, pore sizes are calculated from the air pressures required to clear 'wetted' pores. Large pores need lower pressures to clear while higher pressures are required to clear the smaller pores. A plot of increasing pressure against flow rate can be used to calculate a pore size distribution but care must be taken in interpreting the data from this indirect method.

Challenge testing, on the other hand does not depend on second order effects. The filter medium is 'challenged' by a range of particle sizes. The larger particles that pass the filter reflect the larger pore sizes. The primary data from this method is the filter cut point, although in certain cases it is also possible to obtain pore size distributions. The advantage

of challenge testing is that the results are traceable to international standards of length.

### Who is right?

In line with any analytical technique, both Porometry and Challenge Testing methods have sources of error, which must be understood in order to give meaningful results.

In theory, there could be as many answers for a given filter medium as there are Porometers so it is a legitimate question to ask 'What is the correct answer'. A popular method of calibrating a Porometer is to use Electroformed sieves. These are almost

perfectly formed round or square apertures in a thin nickel foil. The 'pores' are accurate to less than a micron and are available in sizes from a few millimetres down to a few microns. It is not too difficult to obtain very accurate porometric results in such idealised filter media but problems begin to occur as the structures become more complex.

Variations in results from Porometers arise from the fact that the theory used to calculate pore size from pressure drop is based on cylindrically shaped pores. Deviations from the model will inevitably reflect in the results, especially in some complex non-woven media.

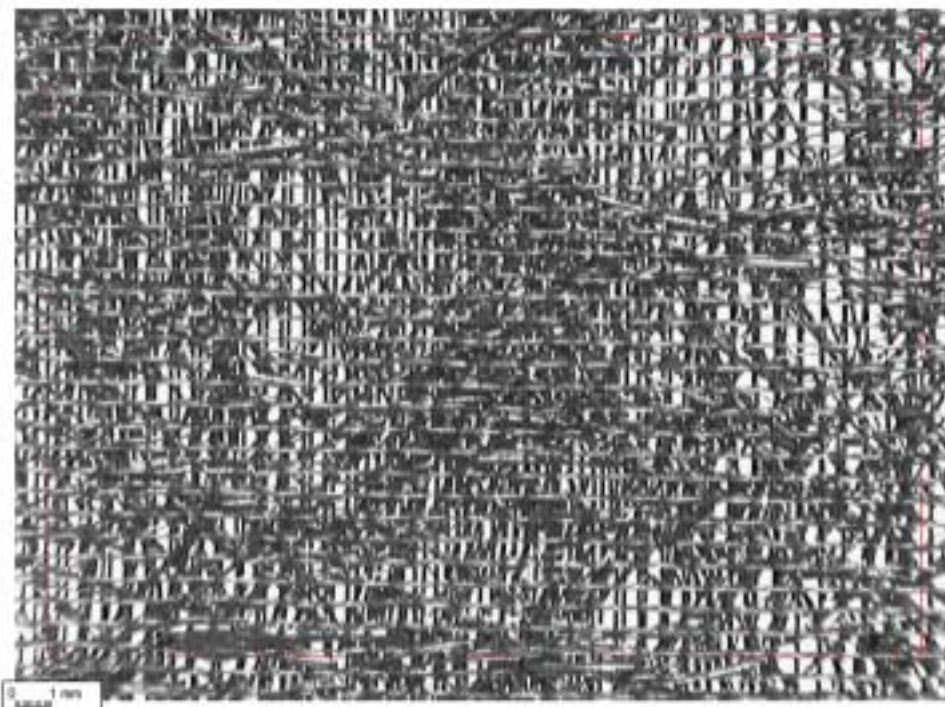


Figure 1. The inhomogeneity of a non-woven stainless steel sand screen

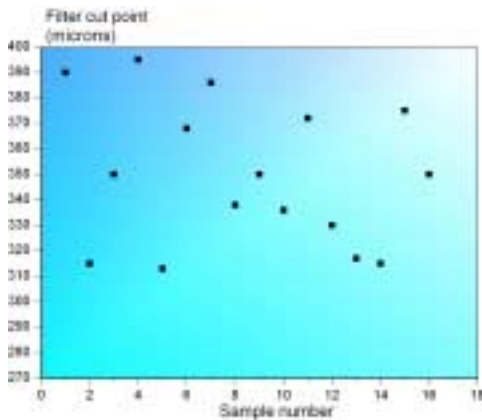


Figure 2. The variability in filter cut points of some non-woven sand screens

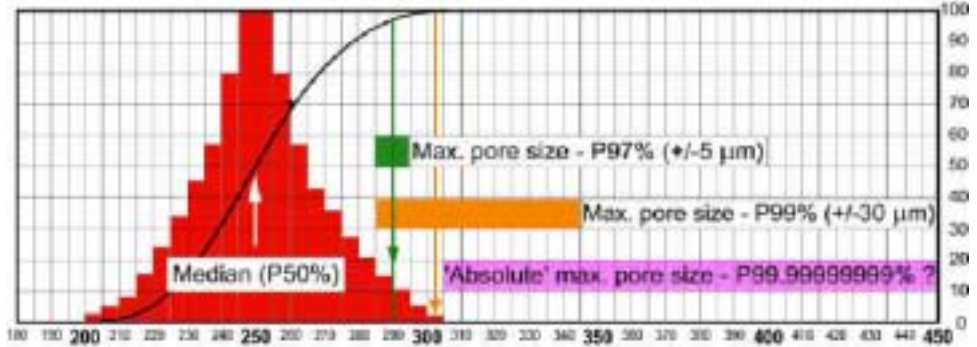


Figure 3. Various definitions of 'maximum' pore sizes and their associated uncertainties

In the case of Challenge Testing, the results can be affected by the particle size distribution and the shape of the challenging particles. When attempting to measure maximum pore size, there is also the possibility of cross contamination, unless working in sterile conditions (see later).

**Repeatability or reproducibility?**

Repeatability or reproducibility are often confused. In general terms, repeatability is the ability of an instrument with its operator to obtain the same, or very close result, when repeatedly testing the same specimen.

Reproducibility however, refers more to the methodology as a whole and is concerned with the errors associated with the generic type of instrument, irrespective of the manufacturer or operator. Consequently error bars on reproducibility measurements are much larger than those obtained from repeatability testing.

In the case of particle size analysis by laser diffraction for example, the reproducibility of the method has been assessed by sending reference standards to up to 70 laboratories having a range of laser instruments made by different manufacturers. Only then is it possible to measure the significance of a result obtained by the technique.

**Uncertainty of measurement**

It is important to note that any measurement of length quoted without an uncertainty is neither statistically nor scientifically valid and can be challenged. For example, a 1000 micron pinhole could be +/-200 microns using a desk ruler or +/- 1 micron using a powerful microscope. Depending on the application, the difference could be critical.

Measurement uncertainty is essential in comparing two sets of data. In the above example, if two pinholes, one 800microns in diameter and the other 1200 microns in diameter were circulated to offices for measuring with a ruler, the answer would come back that they

were identical, but if given to the microscopy department behind the office, they would be significantly different.

Similarly, in pore size measurement, it is not possible to say whether two measurements are significantly different unless the measurement uncertainty is known. Take, for example the inhomogeneous non-woven sand screen filter in figure 1.

Results could be up to 27% apart because of the random method of construction of the medium, figure 2. Therefore, statistically a 27% deviation in results would be insignificant for the specimen!

By contrast, in a precision woven sand screen, only results greater than approximately 5% apart are considered significant.

**Maximum pore size**

Maximum pore size is a much abused parameter in the filtration industry in that, without qualification it is almost meaningless. For example, 'absolute' maximum pore size is the single largest detected pore, but how far must one look to find the pore, a single 25mm disc or several hundred square meters on a roll of the filter media? As seen above, it is much easier to find a representative sample of a woven filter than its non-woven counterpart.

The reliability of the maximum pore size measurement is therefore a function the homogeneity of the filter media as a whole and the ability to take a representative sub-sample for analysis.

Assuming that a representative sample can be taken, the

confidence in the maximum pore size is dependent on the number of pores examined. There is less uncertainty in finding and measuring 1 in 100 pores (P99%) than in finding and measuring 1 in 100 million (P99.99999999%). Measuring a maximum pore size of P97% where there are 3 in 100 pores is far more certain and so has a narrower uncertainty band, figure 3.

In Porometry, where the absolute maximum pore size is based on the appearance of a single bubble through the wetted filter medium, the uncertainty is relatively high because the answer depends the homogeneity of the specimen and the ability to find the maximum pore.

**Table 1 – Pore size comparisons for Madison DualTex™ woven filters**  
(Porometer data courtesy of E Mayer, Du Pont, USA)

Filter code	PX-20	PX-40	PX-50	PX-70	PX-100	PX-140	PX-170	PX-200
Sonic Cut point (efficiency at 97%)	21	36	48	75	95	140	175	203
Coulter efficiency <sup>1</sup> (98%)	21	33	53	106	94	135	176	176
PMI efficiency <sup>1</sup> (98%) (a)2004 data	34	60	74	140	101	141	260	213
(b) 2006 data	22	36	47	106	97	138	180	206
PMI air perm(cfm/ft <sup>2</sup> ) (2004 data)	1.3	8	13	97	29	65	285	180
1. Bubble point / 1.7 (Tortuosity factor)								

Similarly in challenge testing, the absolute maximum depends on finding a single particle out of several million that may have passed the filter. It is disappointing that so many technicians point to one particle on a microscope image and declare confidently that there must be a pore that size in the filter. However, unless the experiment was conducted in clean room conditions, it could have settled there from the atmosphere.

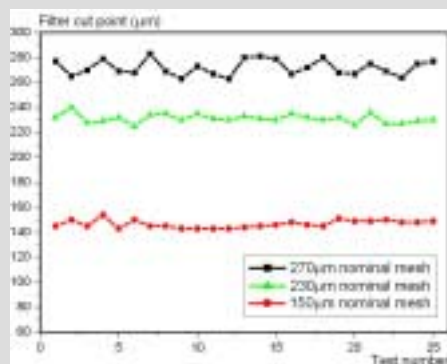
### Precision weaving of sand screen for the oil industry

United Wire Ltd of Edinburgh, Scotland is a well established wire weaving company which has been producing stainless steel cloth for many years. Measuring the pore size of plain weaves is a comparatively easy task because the apertures can be viewed by transmitted light.

In the case of sand screens, however, the filters have to be significantly stronger in order to have the necessary mechanical strength required to place them up to 10,000 meters under the seabed. The complex weaving required renders the cloth opaque to light, making microscopic measurement of the pore sizes impossible.

United Wire turned to Whitehouse Scientific to measure the pore sizes of their sand screens. The new Sonic Challenge test method involves challenging the filter with narrow size distribution glass calibration beads fluidised on the surface of the filter using pulsed acoustic energy. The filter cut point can be derived simply from the percentage of the microspheres passing.

The precision of the weaving process and the Sonic Challenge test is illustrated in the graph below showing that, over a prolonged production run, the variation in filter cut point is less than 5%.



Sonic Challenge tests for sand screen production. Variations are less than 5%.

### Comparing results

By using a term known as the 'tortuosity factor', bubble points from Porometers can be converted into an equivalent value to the filter cut point obtained from challenge testing. It is then possible to compare the P97% maxima from several pore size measuring instruments. This was done for a complex 3-dimensional woven filter cloth from Madison Filter, figure 4. The results are shown below.

As the process control was based on the Challenge test, it was not surprising that the nominal and measured sizes were very similar for the Sonic cut point method. The Coulter Porometer gave reasonable results with one or two exceptions. In the original 2004 PMI data quite a few anomalies were seen and so the tests were repeated in 2006. This produced a much improved data set but PX-70 was still almost 50% higher than expected, but agreed very well with the Coulter result. Air permeability measurements showed that this specimen had a comparatively high porosity, which could have distorted the Porometer measurements.

### Conclusion

When measuring filter cut point or pore sizes, it is very important that the uncertainties of the methods are known and preferably, stated, otherwise it is not possible to tell whether a difference between two measurements is significant or not.

This is particularly important in the petrochemical business for certain non-woven filter sand screens where variations in cut point in a cloth can be as high as 30%.

Absolute maximum pore sizes should never be used except in certain clinical applications where the passage of a single virus could be catastrophic. The 97<sup>th</sup> percentile of the pore size distribution is a more statistically robust measure of 'maximum' pore size.

Significant differences in results between Porometers have been seen, raising a question mark over the reproducibility of the technique as a whole. Unlike the simplicity of the challenge test method, Porometers are second order instruments relying on theory and software to obtain the best results, which make them less user friendly. Whether better user access to the software could improve matters remains to be seen, for example, changing the tortuosity factor in line with porosity. It would be very interesting to conduct a similar round robin to that in the particle size analysis area to finally quantify the universal uncertainty of measurement of this technique.

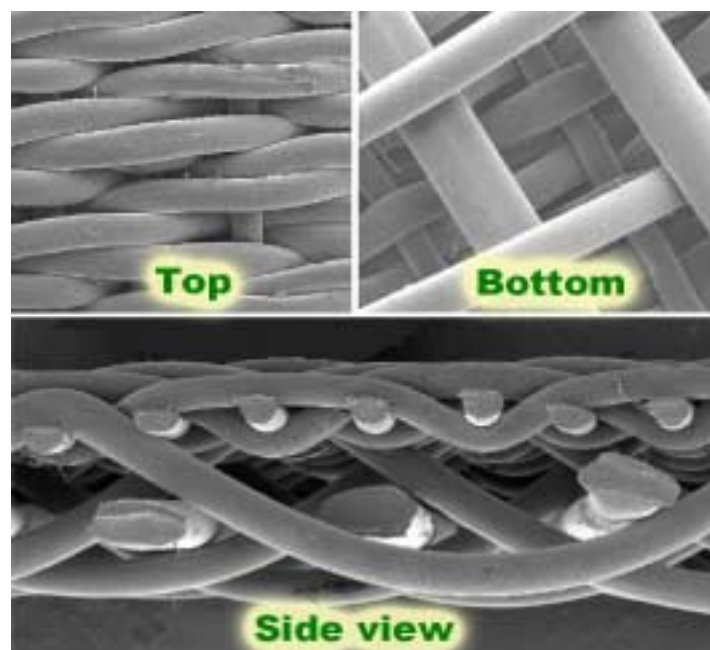


Figure 4. A complex woven filter used in comparing Porometers and a Challenge Test method of pore size analysis (Courtesy of Madison Filter)