Analysis and monitoring:

how to improve precision pore size measurement

In this month's column, Dr Graham Rideal introduces us to the benefits of 'challenge testing' by precision glass microspheres, and explains why this is an area companies should take an interest in.

The filtration and separation business has a worldwide potential of over £20 billion – but to be most successful in obtaining a share of the action, increasing demands are being made on quality assurance measurements. Although second order methods – such as Porometry – are increasing in accuracy and repeatability, the real test of filter efficiency is the ability of a medium to physically hold back real particles. The method, known as 'challenge testing' has an added benefit in that the pore size results can be traced back to international standards of length such as NIST *(the National Institute of Standards and Technology).*

Defining pores

Unfortunately, when a filter pore size is quoted, two assumptions are made, firstly, the pores can be described by a single parameter (for example, they are circular); and secondly, they are all of the same size.

An ideal type of filter is an electron-etched foil where the apertures are circular and have the same size. Only in this case can pore size be described by a single parameter – pore diameter. All other filter media are irregular 3-dimensional structures whose pore size, shape, depth and size distribution is not so easily described (*see figure 1*). Although complex mathematical modelling can give insight into pore structure, it is important not to lose sight of the function of a filter, which is to clarify a liquid or gaseous suspension of particles.

Notwithstanding pore size definition, the two most common specifications of a filter

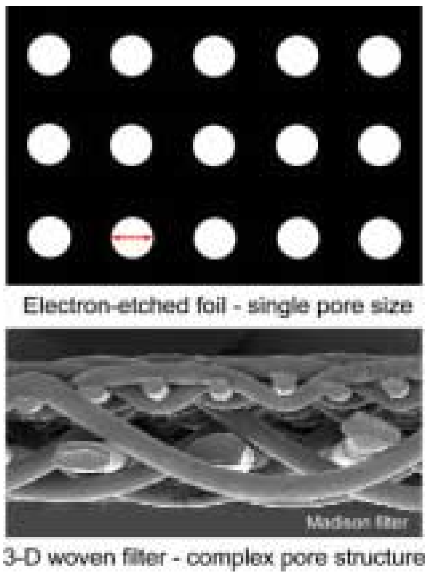

Figure 1: pore sizes are not easy to define in complex filters

are **mean** or **median** size (sometimes called the nominal rating) and the **maximum pore** size. Mean or median sizes are conceptually easy to understand in that they are simply averages of all the pore sizes, however the relationship to the performance of a filter is a much debated issue.

Maximum pore size is more relevant to the ability of a filter medium to clarify a suspension and may be the foremost parameter sought in a filter but without qualification the term can be very confusing. For example, 'absolute' maximum pore size is the single largest detected pore, but how far must one look to find the pore?

In fact, the 'absolute' maximum pore size can only be found in a 100% examination of all the pores in the final assembled filter system constructed from the filter medium, which is clearly impractical for most applications. Furthermore, welding or other assembly errors in the filter system could introduce flaws that totally eclipse any attempt at measuring the 'absolute' maximum pore size.

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To try to estimate the absolute maximum pore size pore from a small part of a filter can lead to uncertainties so large that the measurement is too unreliable to be of any use. The reliability of the maximum pore size 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 one in a 100 pores (P99%) than in finding and measuring one in 10 million (P99.99999999%) while there is

Table 1: band widths of filter standards (µm)				
16-25	20-34	26-36	31-46	36-55
45-62	53-73	63-86	75-103	80-123
106-147	127-175	151-209	180-248	214-295
252-346	304-417	360-498	383-591	484-700

most confidence in measuring a maximum pore size of P97% where there are 3 in 100 or 30 in 1000 pores. Pore size defined as P97% is therefore considered the most statistically robust method of determining the maximum pore size.

Having a precision range of traceable microspheres is only the first stage in measuring filter cut points. There must also be an accurate and repeatable way of presenting (challenging) the filter medium with the standards.

In applications where the minimum pore size is also important, exactly the same statistical considerations apply as in the case of maximum pore size.

The challenge test principle

Although defining pore size distribution of a filter medium is very useful in terms of its specification for a purpose, a practical assessment of performance should be the ultimate goal; will it retain the target particles?

Defining a filter medium by a more performance-related criterion such as cut point may be more helpful. Cut point is measured by challenging the filter with real particles and measuring the maximum particle sizes passing. For the most unambiguous results, the challenging particles should be spherical and have a narrow particle size distribution.

Pore size ranges of the filter calibration microspheres

In the example above (*see table 1*), over 20 narrow particle size distribution glass microsphere standards have been prepared to cover pore sizes from 16 – 700 microns.

By accurately measuring the particle size distribution both by microscopy and a precision electroformed sieving method, it is possible to construct a calibration graph for the microspheres where the percentage of the beads passing an unknown mesh can be used to calculate the filter cut point

As the results are traceable to international standards such as the *National Institute of Standards and Technology (NIST)*, the derived filter cut points are also NIST traceable.

The challenge test instrument

Having a precision range of traceable microspheres is only the first stage in measuring filter cut points. There must also be an accurate and repeatable way of presenting – or 'challenging' – the filter medium with the standards.

The *Gilsonic Autosiever*, is a unique system that uses intense sonic energy to produce an oscillating column of air, which flows through the body of the mesh. Power 'ramping' and a tapping action minimizes pore blockage by the challenge particles.

Making use of challenge testing by precision glass microspheres can help give an unequivocal measure of performance and is being increasingly adopted by a wide range of filtration and separation companies.

A final word

Although the projected filtration and separation business is enormous, those getting the biggest slice of the cake will be the industries capable of producing (and demonstrating) the highest quality filters. Making use of challenge testing by precision glass microspheres can help give an unequivocal measure of performance and is being increasingly adopted by a wide range of filtration and separation companies.

About the author:
Dr Graham Rideal graduated from Lancaster University and joined ISI where, after 15 years, he became the company consultant in particle size analysis. He is the auther of several patents describing the construction of inorganic materials such as foams, films and coatings from nano-sized mineral particles. He is the founder of Whitehouse Scientific – a company specialising in particle size standards – and is chairman of the Filtration Society, based in the UK.

Figure 4: fabricated sand screen tubes showing weld lines

Results in critical applications

One of the most costly errors resulting from inaccurate pore size measurement can occur in the oil industry where filter 'plugging' under the sea bad can result in a £20 million bill to relocate the oilrig and redrill another 10 km piple line. As a result, challenge testing using precision glass microspheres has now been adopted as the most adopted as the most accurate and repeatable method of measuring sand screen performance. As part of the quality assurance, the sand screen tubes (*see figure 4*) are cut into six 90 mm discs and measured.

The results in table 2 show the consistency.

Sample	1	2	3	4	5	6
% passing	27	28	27	31	26	28
Cut point (µm)	138	139	138	140	138	139
Filter standard used 127-175 (µm)						

Table 2. checking the consistency of the cut point in a 115µm sand screen tube