



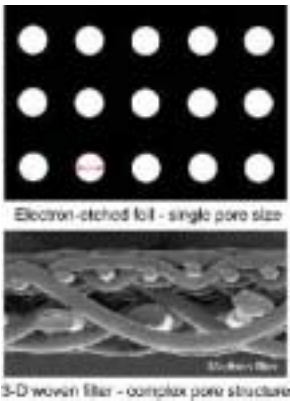
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# Measuring Filter Performance by Challenge Testing

## Part 1: Particles and Apparatus

### Defining Pore Size

An idealised form of a 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, 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.

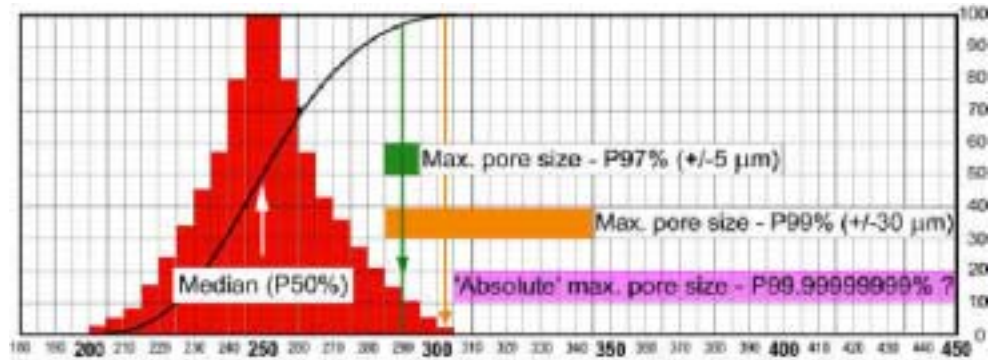


**Figure 1.**  
Pore sizes are not easy to define in complex filters

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 has more relevance 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’

Notwithstanding pore size definition, the two most common specifications of a filter are mean or median size (sometimes called the nominal rating) and the maximum pore size.



**Figure 2.** The definition and reliability of 'maximum' pore size measurements

maximum pore size is the single largest detected pore, but how far must one look to find the pore?

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. 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 1 in a 100 pores (P99%) than in finding and measuring 1 in 10 million

(P99.99999999%). Measuring a maximum pore size of P97% where there are 3 in 100 or 30 in 1000 pores is far more certain, figure 2.

Pore size defined as P97% is therefore considered the most statistically robust method of determining the maximum pore size.

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

### Choosing Challenge Test Particles

Although defining the pore size distribution of a filter medium may be very informative, a practical assessment of performance should be the ultimate goal; will it retain the target particles? In any case there are limited methods of measuring pore size distribution, for example Porometry and Porosymmetry, and even then the results are often instrument dependent.

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

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most unambiguous results, the challenging particles should be spherical and have a narrow particle size distribution, figure 3.

In preparing challenge test particles, the ideal width of the particle size distribution should be about 3 sieves in the ASTM or ISO series, for example 53 – 75 microns. This enables 3 sieves to be used in the certification process i.e. 53, 63 and 75 microns, see later.

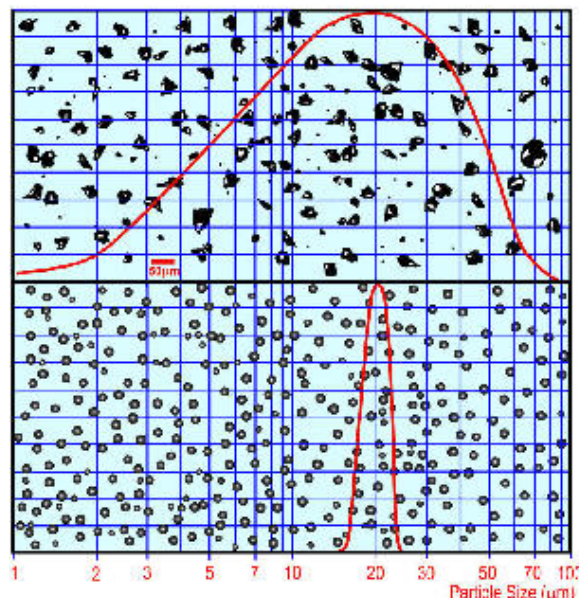
**Preparing Identical Samples for The Challenge Test**

One of the most difficult and often overlooked aspects of calibration standards is in the preparation of identical sub samples from a master batch of powder. Unless the powder has an ultra-narrow particle size distribution there is always the possibility that some settling or segregation of particles of different sizes can take place. A consequence is that, not only is the small sample extracted not representative, but the remaining powder in the larger bottle has also been changed.



**Figure 4.**  
A 100 stage spinning riffler produces identical sub-samples

To overcome sampling problems it is essential that sub samples are taken in a representative way. The most effective method of subdivision is a spinning riffler where a carousel of bottles is rotated beneath a flow of powder from the master batch. Depending on the number of revolutions, each bottle can contain



Upper: A Test Dust. Lower: Spherical Standards

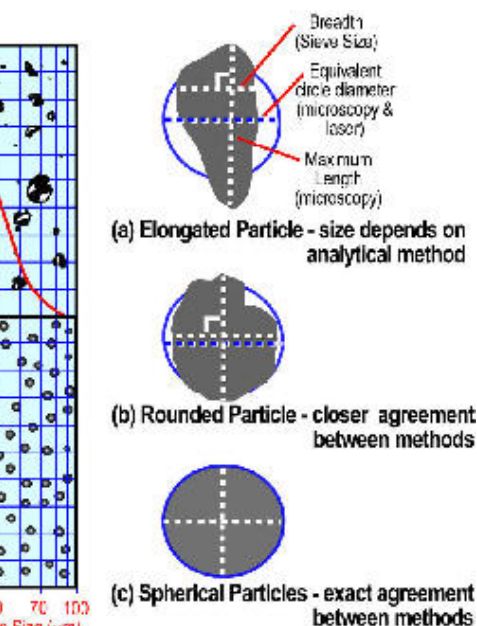
**Figure 3. Spherical, narrow size distribution challenge particles give accurate and unambiguous results**

several hundred portions from the main sample, making each sub sample an identical fraction of the master batch, figure 4.

**Certifying Challenge Test Microspheres**

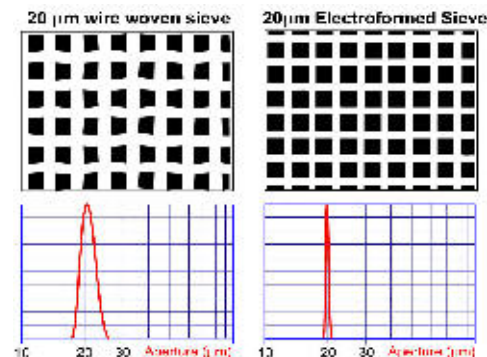
Having produced identical samples, the next most important issue is ensuring that the particles are certified by a parameter that will reflect pore size. For example, in an extreme case of particle shape where the particle more closely resembles a fibre rather than a sphere, measuring the fibre length will not reflect pore diameter. The fibre diameter is a much more relevant parameter. When certifying microspheres used as filter standards therefore, a sieving method, which measures particle width, will more closely represent pore diameter, see figure 3. Note. As glass microspheres are formed by a melt process, there is always the possibility distortion taking place during cooling. Because sieve analysis measures width, the results will not be affected by small changes in sphericity.

Sieve analysis however is only as accurate as the sieves used in the test. Contrary to a popularly held belief, wire woven sieves do not all have identical apertures and the weaving process can



give rise to a wide variation of openings. These are specified in both ISO and ASTM standards. Wire woven sieves cannot therefore be used as reference sieves for certifying reference standards. Instead, high precision Electroformed sieves must be used. In these sieves, apertures vary by less than 2 microns so they are ideal for calibrating challenge standards, figure 5.

Electroformed sieves are available with aperture sizes down to 5 microns but, although the manufacturing tolerance of 2 microns is excellent at the higher apertures (standard aperture sizes go up to 2000 microns), the variation becomes significant below about 10 microns. Certification of challenge test



**Figure 5.**  
Comparison of wire and Electroformed sieves (micrographs in negative for image analysis)

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microspheres by Electroformed sieves is therefore limited to a minimum size of 15 microns. For smaller sizes, microscopy and image analysis must be used.

The Electroformed sieves are certified by optical microscopy using a reticule calibrated by NIST to give traceability to the International standard of length.

In the certification process, each filter standard is tested 5 times on 3 Electroformed sieves in a Gilson Sonic sifter capable of analysis down to 5 microns in the dry state (see later). The measurement uncertainty is then determined. To ensure that there is a smooth transition of sizes throughout the range, the data is backed up by microscopy. This gives confidence in interpolating the data from just 3 points from the Electroformed sieve analysis data, figure 6.

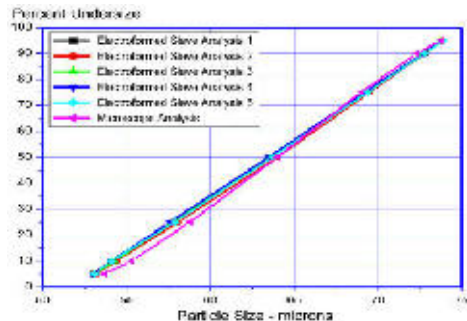


Figure 6. Certification data from a 53 – 73µm challenge test standard

Traceability is therefore transferred from the NIST reticule to the calibrating glass microspheres. Thus using the certified standards to test a filter will confer NIST traceability to the unknown pore size filter.

## Measuring Filter Cut Points

### (a) From 1000 – 15 microns

The particle size distribution graph of the challenge particles, figure 6, is then redrawn to express the measurement uncertainty but now, instead of knowing the aperture size of the Electroformed sieves and measuring the cumulative percent of the microspheres at the various sizes, we know the cumulative percent of the microspheres and, from the percent passing, we can determine the aperture size in the filter, figure 7.

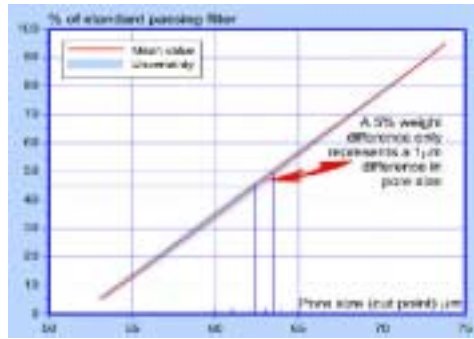


Figure 7. A calibration graph for measuring the pore size of a filter

It can now be seen why narrow particle size distribution standards give such accurate results. A 5% difference in the weight passing a filter only corresponds to a 1 micron difference in the pore size (cut point).

To determine the cut point, a 90mm disc of the filter is cut and mounted in a Gilson Sonic sifter, figure 8. About 0.2g of the appropriate filter standard is then fluidised by rapidly oscillating air currents on the surface of the filter for about 1 minute and the percentage of the standard passing is calculated. The calibration graph, or the corresponding mathematical formula, can then be used to determine the cut point.

The range of calibrating microspheres for this dry testing method is shown in table 1.

Table 1: Band Widths of Sonic 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

### (b) Measuring Filter Cut Points Below 20 microns

Below about 20 microns a combination of particle-particle attraction forces and air permeability through the filter restricts the sonic fluidisation process and an aqueous system must be used to permeate the filter with the challenge particles. In order not to create a secondary filter on the surface, dilute suspensions must be used.

A range of narrow particle size distribution filter standards from 20 microns down to 2 microns has been prepared for the suspension challenge test, table 2.

Table 2: Band Widths of Suspension Filter Standards (µm)

2-6	5-9	8-16	12-22
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Figure 8. A Gilson Sonic sifter for filter testing with filter holders

These standards are traceable to NIST and are made from the highest sphericity glass microspheres. They are too small to be certified by Electroformed sieves so are calibrated by high resolution optical microscopy capable of resolving to 0.1 of a micron. Particular attention has been paid to corrections that must be applied to compensate for the diffraction effects of such small particles (Space precludes a detailed description in this article).

The simplest challenge method is to draw the calibrating microspheres through a disc of the filter under vacuum on a Buchner flask, figures 9 and 10. In this apparatus approximately 100mg of the calibrating microspheres in 20ml of water are used. The particle size after the filter is then compared to the original particle size distribution of the original filter standard.

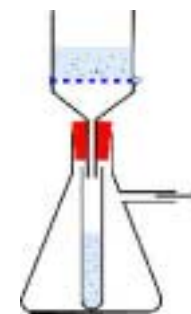


Figure 9. The principle of suspension challenge testing



Figure 10. Clarifying a filter standard in a suspension challenge test apparatus

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Although a flat disc is the most convenient geometric form for testing a filter, holders have been made for conical and cylindrical filters, figures 11 and 12.



Figure 11. A filter cone holder for challenge testing (wet or dry)



Figure 12. A challenge test apparatus designed for testing air filters (mounted on an Electromagnetic shaker)

## Challenge Test Results

### (a) Sonic filter tester

To understand the cut point more precisely, one must analyse the particle size distribution of the standard after it has passed the filter and compare it to the cut point, figure 13.

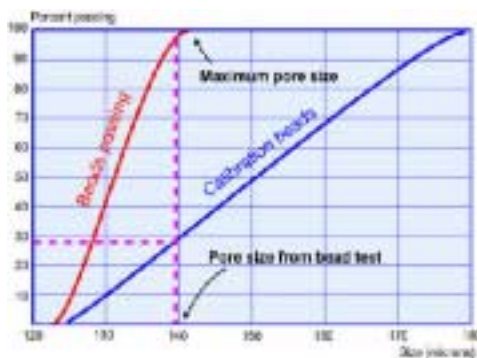


Figure 13. Microscope analysis of the beads passing compared to the sonic test result

In figure 13, 29% of the standard passed the filter, which equates to a cut point of 140 microns. When the microscope analysis of the standard passing the filter is superimposed on the calibration graph, the cumulative percent undersize at 140 microns corresponds to a percentage of approximately 97%. This suggests that the filter would be 97% efficient in

trapping particles above 140 microns. In other words, the cut point corresponds to a pore size close to the maximum pore size in a filter.

### (b) Aqueous suspension challenge tests

In this method, the filter is challenged initially with the larger size filter standards. The cloudy suspension should be clarified, figure 10. As the particle size of the standard is reduced more will pass the filter and analysis of the partially clarified suspension can be used to determine the cut point, figure 14.

It can be seen that the nominal 10 micron filter (used in inkjet printers) is very effective in excluding particles over about 9.5 microns.

It is interesting to note that nominal rating of a filter does not always work out in practice. One air filter having a nominal rating of 5 microns, when tested had a cut point of 140 microns!

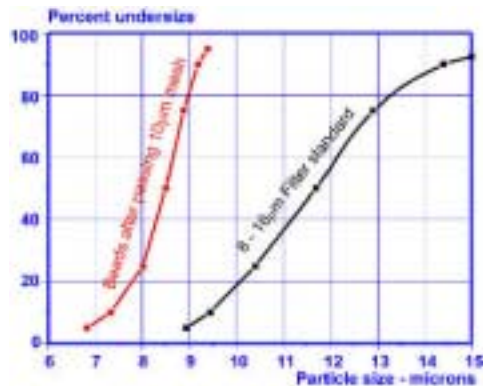


Figure 14. Results of challenge testing a 10 micron filter (aqueous suspension method)

## Conclusion

Part 1 of this two part study of challenge testing has majored on the basic principles of the method and investigated new developments that refine the technology. One of the biggest advantages of the method is that it produces absolute and unambiguous results that can be traced back to the International standard of length. Furthermore, it is conceptually easy to understand: calibrated microspheres either pass or are trapped by the filter.

Indirect pore size determinations such as Porometry (bubble point) and Porosymmetry (liquid intrusion) rely on modelling pore shape so can lead to significant differences in pore sizes, particularly for highly complex pore shapes or pore sizes above about 100 microns.

Preliminary results have sought to understand the relationship between cut point and pore size. In summary, the cut point is the size above which about 97% of suspended particles are trapped by the filter. In practical terms the cut point reflects the ability of a filter to clarify a suspension.

The inability to predict filter performance can have dire consequences in many industries. These early results there have shown that there can be significant differences between the claimed pore size and the actual maximum pore size.

Part 2 of study will be a more in depth investigation of the data than can be achieved from challenge testing including pore size distribution determinations and will make comparisons of pore size measurements in critical applications.

## About the Author

Dr. Rideal graduated from Lancaster University, England. He is the author of several patents describing the construction of inorganic materials such as foams, films and coatings from nano mineral particles. He was the founder of Whitehouse Scientific in 1983, a company specializing in particle size standards, which was selected as the top certification laboratory by the European Bureau of Certified Reference. The election of Dr. Rideal to the position of Chairman of the Filtration Society recognizes his unique contribution to developments in the field of pore size measurement of filter media. Dr. Rideal has recently joined with other prestigious members on Filtration News' Editorial Advisory Board.