# CALIBRATION OF FILTER MEDIA USING NIST TRACEABLE REFERENCE STANDARDS

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#### Introduction

Simple wire woven meshes such as test sieves are not difficult to calibrate. They are essentially 2-dimensional structures that can be easily calibrated by microscopy. Complex 3-dimensional weaves, however, are opaque and so cannot be calibrated by microscopy. Although bubble point testing has been used to determine aperture or pore sizes, the technique is not a direct method and begins to lose accuracy above sizes of about 50 microns.

One of the few absolute calibration methods available for filter media in the size range  $1-500\mu m$  is based on the permeability of test dusts. However, the accuracy of this method is limited because test dusts are irregular in particle shape and have a broad particle size distribution. The combined effect results is an uncertainty both in the true aperture width and the resolution of the method. To overcome these difficulties, a range of about 20 narrow distribution microsphere standards has been developed to accurately determine aperture size. The advantage of using spherical standards compared to irregular particles is clearly seen in figure 1.

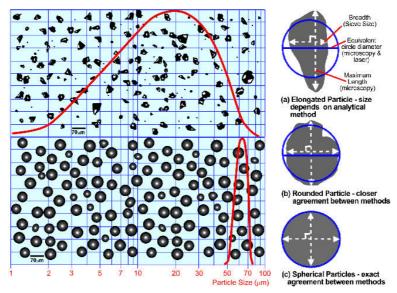


Figure 1.

TopLeft: BelowLeft: Right: Size distribution of a typical test dust.

Narrow size distribution of filter calibration microspheres.

Size definitions of irregular particles.

### **Production and Certification**

Broad distribution glass microspheres from 5 to over 500 microns have been fractionated into narrow size distributions. The master-batches of the individual grades were then subdivided into 'single-shot' bottles containing from 0.1 to 0.5g of microspheres depending on the calibration range of the standard. The 100 stage spinning riffler used has been independently assessed by the European Community (Bureau of Certified Reference), Malvern Instruments and Loughborough University (UK). The results show an insignificant variation in particle size from bottle to bottle so any difference in aperture size must be a function of the medium being tested and not the microspheres used in the test.

In the certification of the calibration microspheres, highly accurate electroformed sieves down to 5 microns were used so that a breadth related size was measured (see the effect of shape on size definition in figure 1). This is the most appropriate parameter for the passage of a particle through a filter medium. The electroformed sieves were calibrated by optical microscopy and image analysis using a National Institute of Science and Technology (NIST) traceable reference graticule. The microspheres measured on the sieves were thereby NIST traceable.

The Electroformed sieve analysis data for the microsphere standards was then used to construct a calibration graph to determine the aperture size of an unknown filter medium, figure 2.

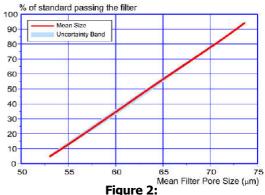
The advantage of having a very narrow particle size distribution is now clearly seen as, in the graph of the  $50-75\mu m$  microsphere filter standard, weight variations of as much as 5% in the percentage of the standard passing results in aperture size variations of less than  $1\mu m$ .

## **Calibrating filter media**

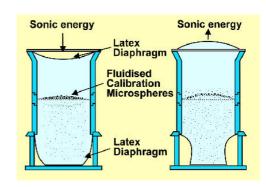
A high performance sonic method has been developed for calibrating filter media. Conventional sieve shakers, as the name implies, shake the sieves, but sonic sifters move a column of air at high speed through the sieves or filters at frequencies of up to 3600 cycles per minute so the particles rather than the sieves are shaken, figure 3.

Such intense energy overcomes inter-particle attraction forces so that, unlike conventional sieving where dry sieving is only effective at sizes above about  $50\mu m$ , sonic sieving has the capability of separating individual particles right down to  $5\mu m$ , however, for low air permeability samples, a wet suspension method may still be required for aperture sizes below about 20mm. The sonic filter tester is shown in figure 4.

To measure the aperture size of a mesh, a 90mm diameter disc is clamped into a clear plastic frame. About 0.5g of an appropriate filter calibration standard is then weighed onto the filter and fluidised on the sonic sifter for about 1 minute. From the percentage of microspheres passing the filter, the aperture size can be determined from the calibration graph supplied with each standard. The aperture size is defined as the size above which less than 0.1% per minute of the standard passes the filter medium.



Calibration graph from a Certificate of Analysis



**Figure 3:** Sonic action in the filter tester



**Figure 4:** The sonic filter tester

#### **Quality assurance**

As this method is so accurate, it can be used to check the consistency of filters during manufacture. For example, a tight cluster of samples can be analysed from a localised area of a woven mesh to determine the error bar for the particular mesh. Remote specimens can then be taken to determine if the aperture size falls outside the error bar. Table 1 shows the repeatability on a nominal  $75\mu$ m plastic woven mesh. The standard deviation was only  $2\mu$ m.

**Table 1.** Repeatability test using a plastic woven mesh

Sample no.	Initial standard weight (g)	Weight after testing (g)	% of standard passing	Aperture size - µm
1	N 448	N 2N7	53 R	76 N
2	0.459	0.170	63.0	78.3
3	0.454	0.146	67.7	79.6
4	0.412	0.191	53.7	76.0
5	0.431	0.225	47.8	74.4
6	0.421	0.233	44.5	73.6
7	0.407	0.176	56.7	76.8
8	0.415	0.206	50.3	75.0
Average aperture size = 76.2um (Standard deviation = 2.0um)				

This novel method has several advantages for the filter manufacturer:

- 1. New product development is considerably simplified
- 2. Large production runs for field trials can be minimised
- 3. Certifying a filter to NIST standards gives customers confidence in the manufacturers technical ability
- 4. The return and replacement costs of off-spec filtration products can be virtually eliminated

## Oil drilling application

Precision calibration of filters is especially important in the offshore petrochemical industry. In order to eliminate the pumping of sand from the oil beds, high performance stainless steel meshes must be inserted at the extraction point deep on the seabed. Failure to extract the sand results inconsiderable wear from the highly abrasive sand and oil slurry, not only in the pumping gear, but also in the pipe below ground. The financial consequence of a rupture in the conducting pipe work is enormous because the oil rig would have to be moved and a new bore hole sunk. A typical cost would be upward of US\$ 30 million.

Many of the major oil companies have now specified the glass microsphere method of calibrating their sand screens not only because of the speed and accuracy, but because the aperture sizes produced are directly traceable to the international unit of length.

An additional advantage of using glass microspheres for calibrating filters is that the fraction of the standard passing the filter can be analysed by microscopy so that the absolute maximum aperture size can also be readily determined.