Calibration of sub-20 micron filter meshes

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1. Background

Whitehouse Scientific produces a unique range of dry filter standards for calibrating filter meshes having mean apertures from 18 to over 500µm. Sonic energy is used to transport the glass microsphere standards through the meshes (see 'Library' in www.WhitehouseScientific.com)

The advantage of using solid glass microspheres is that absolute or primary results can be achieved, ie, the aperture size does not require interpretation from a second order effect such as bubble point or air pressure tests – the beads either pass or they do not and those passing can be measured directly by microscopy.

Below 18µm inter-particle attraction increases to such an extent that it becomes very difficult to transport the individual microspheres through what is a tortuous path of apertures in the body of complex filter meshes – the particles do not fluidise under the influence of the intense sonic energy. A liquid based method has therefore to be employed. This short report describes the development of a new microsphere suspension technique, which can extend the lower limit of filter calibration to below 5µm. Pivotal in the methodology is the availability of well defined glass microsphere standards in the range 3 - 25µm.

2. Experimental method

2.1 Apparatus

0.1g of the calibrating microspheres were put in a 50ml beaker with 1 drop of dispersant (Ultrasonic cleaning fluid). The suspension was then placed in an ultrasonic bath for 2 minutes to ensure complete dispersion.

The filter mesh to be calibrated was placed in the filter holder as described opposite (in this case a complex laminated system from G Bopp of nominal aperture rating below 10µm)

The suspension of the calibrating microspheres was poured onto the filter mesh under test and, using a Buchner flask to provide a vacuum, the suspension was sucked through the filter mesh into a collecting test tube for analysis by microscope. An ultrasonic probe was used to disperse the microspheres on the filter.

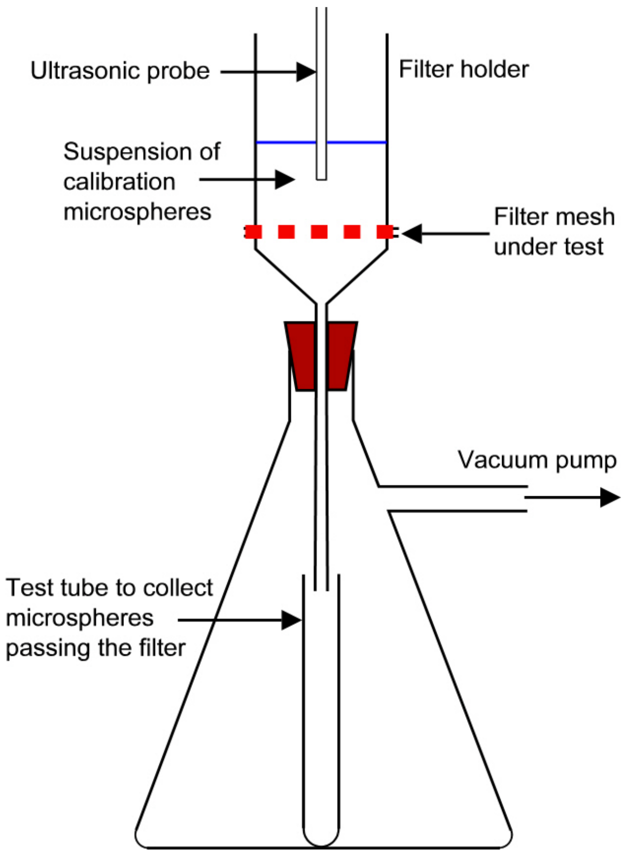

Figure 1. Filter calibration apparatus – wet method

2.2 Calibration microspheres

Three overlapping polydisperse calibration standards were used to determine the aperture size in the filter mesh.

The first one selected was a BCR 'Mirror' standard (see web site) having a size range between 2 and 7µm peaking at 4µm – nominally 1 - 10µm.

The second was between 10 and 20µm while the third, an intermediate grade was between 8 and 16um.

Finally, a monodisperse polystyrene latex of 8µm was used.

2.3 Microscopy and image analysis

The Whitehouse Shapesizer Image analysis system was used to determine the size distribution of the beads passing the filter mesh. The data can be traced directly to the International unit of length. Guidelines for the methodology can be found on the company website.

Figure 2. Whitehouse Shapesizer Image Analysis System

3. Results

3.1 Using a 2 - 7µm microsphere calibration standard

The suspension passing the filter into the collection test tube was as milky as that introduced into the filter holder suggesting that all the particles had passed. The particle size analysis confirmed that there was no difference in size between the filtered and the feed suspension.

The aperture size was therefore greater than 7 microns.

3.2 Using a 10 - 20µm microsphere calibration standard

In this experiment, the liquid passing was totally clear showing that the filter had removed all particles above 10µm.

On the evidence of the first two tests therefore, the final aperture size was expected to be between 7 and 10µm.

3.3 Using an 8µm monodisperse latex standard

About 50mg of the dry powder was placed on the filter in the holder and 50ml of water and one drop of dispersant added. After brief sonication, and with little evacuation, drops of the suspension appeared from below the filter. These were collected and analysed. The results below show that there was little difference in the particle size of the beads after passing the filter.

Table 1. Particle size distribution of 8µm latex microspheres before and after filtration

	Particle size at fixed percentiles						
	5	**10**	**25**	**50**	**75**	**90**	**95**
Before	7.62	7.68	7.79	7.95	8.08	8.23	8.38
After	7.60	7.62	7.76	7.87	8.02	8.14	8.24

The results clearly show that the apertures in the filter are greater than approximately 8.2µm. However the results above show an aperture size below 10µm. An intermediate calibration standard was therefore required which covered the size range 8 - 10µm.

3.4 Using an 8 - 16µm microsphere standard

In the final experiment, an intermediate microsphere standard was used. The results of filtration are shown in table 2 and figure 3 below.

Table 2. Particle size distribution of an 8 - 16µm standard before and after filtration

	Particle size at fixed percentiles						
	5	**10**	**25**	**50**	**75**	**90**	**95**
Before	8.93	9.44	10.40	11.70	12.90	14.40	15.00
After	6.82	7.33	8.02	8.50	8.87	9.18	9.39

Very few particles visibly passed the filter. However, about 10 minutes was allowed for settling in the test tube before they were collected by syringe for microscopic analysis.

The results in figure 3 clearly show the particle size cut produced by the filter. Although 1 or 2 particles were seen up to 10µm, the 95% value is considered a more statistically robust method of determining the aperture size of the filter.

The final size of 9.4µm was consistent with the expected value from the previous tests.

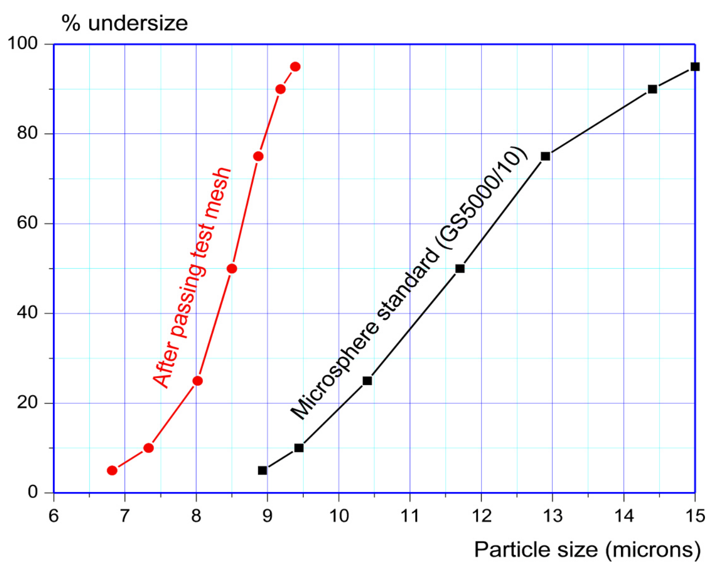

Figure 3. Particle size distribution of an 8 - 16µm microspheres passing the filter

4. Conclusion

Although this wet method of calibrating filter meshes was more time consuming than the Sonic dry method, the results seem very consistent and all the microsphere standards used indicated an aperture size of between 8 and 10 microns. It was not surprising therefore that the final micron rating came to 9.4µm. The final time taken to perform an analysis will probably be about 10 minutes.