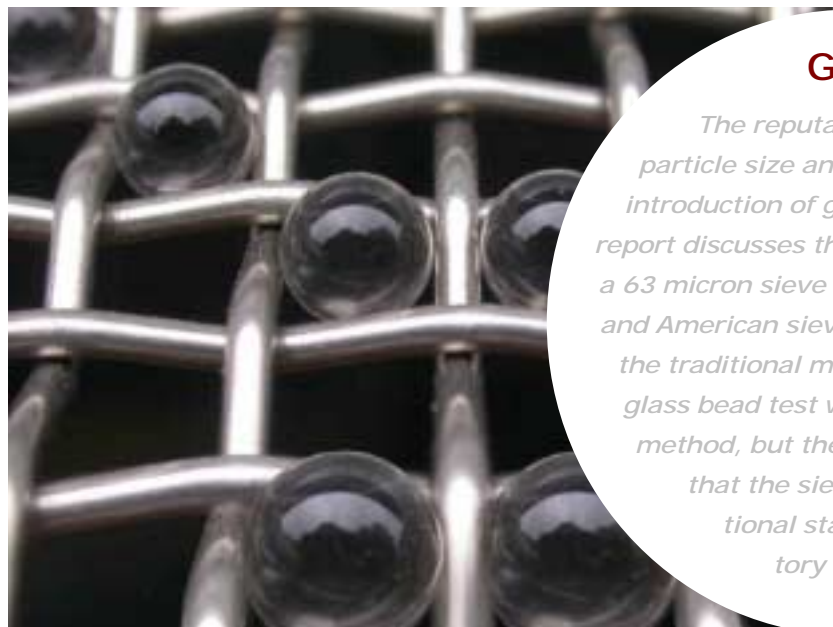




Sieve Analysis – Precision through Calibration



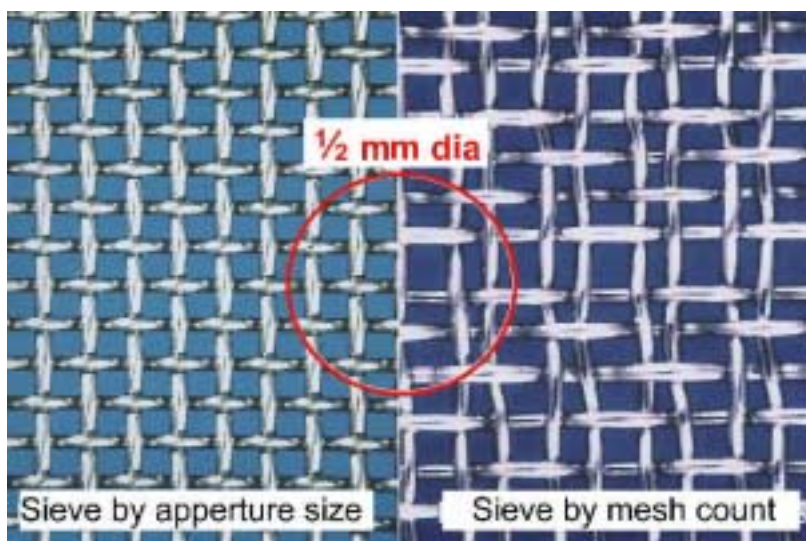
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The reputation of sieving as a method of particle size analysis has been enhanced with the introduction of glass calibration microspheres. This report discusses the results of a round-robin test, where a 63 micron sieve was circulated to the major European and American sieve manufacturers for calibration using the traditional microscope method. Results from the glass bead test were very similar to the microscopic method, but the main advantage for the analyst is that the sieve can be calibrated to international standards in their own laboratory and does not need to be sent away.

Sieving is one of the oldest methods of particle separation and sizing having been used in some form or another since ancient Egyptian times over 5000 years ago. Although often perceived as being old fashioned, in the right hands it can be one of the most accurate methods. The fundamental building block is of course the quality of the sieve apertures themselves. The most common method of construction is to weave the apertures out of fine stainless steel wires. The best weavers are capable of weaving apertures down to 20 microns with a tolerance of +/- 5 microns. For the highest precision, the latest technologies of electroforming and electro-etching can produce sieve membranes having apertures down to 3 microns with a tolerance of better than 1 micron. Unfortunately, there are several newcomers in the market place making claims that simply cannot be backed up by measurement, which is seriously damaging the reputation of sieve analysis. Calibrating sieve apertures has therefore never been so important.

specification of mesh count. When a certain count of wires per unit length is stated, we assume (quite wrongly) that the wires are evenly spaced. Figure 1 shows two woven wire sieves with exactly the same 'mesh number', however, it can be clearly seen that there is a big difference in the size of the apertures between the two sieves. The consequences of using an inferior sieve mesh in a test sieve can be very serious. In the case of the pharmaceutical industry, this could mean lost lives, not just lost money. There is therefore an increasing demand for calibrated sieves, but not just in the pharmaceutical

Fig. 1: Two woven wire sieves with exactly the same 'mesh number' (courtesy of GBopp)



1 The Cost of Getting it Wrong

One method employed to disguise aperture quality is to use the somewhat dated



industry, any industry serious about quality assurance must now show traceability to international standards in their particle size analysis. In this review, a 200mm diameter 63 micron test sieve was circulated to all the major sieve manufacturers for calibration using the ISO and ASTM microscopic methods and the results compared to glass bead calibration methods.

2 Sieve calibration methods

2.1 Microscope

Both the ISO and ASTM methods specify according to wire spacing in the X and Y direction, known as the warp and weft so two sets of data must be collected. The tolerances for a 63 micron sieve is shown in table 1.

The ASTM tolerance rather than the ISO tolerance has been used in table 1 because it more accurately reflects the resolution of the method. The standard simply states that, for a 63 micron sieve, the optical magnification should be between 50 and 500. These days, microscopic techniques are usually accompanied by image analysis software where the pixel resolution is the critical factor.

Not every analysing laboratory specified the pixel resolution, but in no case were the class widths better than 1 micron. It is therefore not statistically justified to quote results to +/-0.1 microns. The worst example was one laboratory that quoted a result for a 2000 micron sieve to 4 decimal places, suggesting a resolution of 0.1 nanometers! What they were actually quoting was an average, not the resolving power of the method.

A second factor often overlooked is the ability to focus on the interweaving wires. At high magnifications it is sometimes impossible to focus on two adjacent wires at the same time because



Fig. 2: Calibration microspheres

the depth of field is too short. As the magnification is reduced to improve focus, the resolution is also reduced.

Calibrating electroformed sieves is much easier than wire sieves because they are flat so resolutions down to 0.1 micron presents no difficulty.

2.2 Sieve Calibration Standards

One of the disadvantages of the microscope method is that the sieves usually have to be sent back to the manufacturer for recalibration. An alternative method, that can be performed in the analyst's own laboratory, is the use of calibration microspheres, figure 2.

In this method, narrow distribution glass beads are certified according to NIST standards using the highest precision electroformed sieves. Because only three sieves are required to cover the size distribution, the results are re-enforced by the higher resolution method of microscopy. This ensures that the beads have a smooth Gaussian distribution, figure 3. Providing the two sets of data can be superimposed, the limited electroformed sieve results can be confidently interpolated to construct a calibration graph with which to determine the mean aperture size of an uncalibrated sieve.

Table 1. The ASTM tolerance for a 63 micron sieve

Nominal aperture Size (microns)	Mesh number	Tolerance at mean	Maximum single aperture	Microscope count
63	230	59 - 67	89	2 x 250

Table 2. Glass bead repeatability test (Endecotts 63 micron test sieve Serial no. 5626574)

Test/technician	Initial Weight (g)	Weight Passing (g)	% passing	Aperture Size (µm)
1/JS	1.01	0.42	41.6	61.7
2/JS	1.14	0.48	41.2	61.8
3/JS	1.15	0.48	41.7	61.7
4/JS	1.08	0.45	41.7	61.7
5/JS	1.09	0.46	42.2	61.8
6/GR	1.13	0.47	41.6	61.7
7/GR	1.11	0.47	42.3	61.9
8/GR	1.17	0.49	41.9	61.8
9/GR	1.10	0.47	42.7	61.9
10/GR	1.10	0.47	42.7	61.9
Mean size 61.8 +/- 0.2µm				

2.3 The Near Mesh Method

The final method of calibrating the sieve was the so-called 'near mesh' method. Glass microspheres that are almost identical in size to the apertures firmly lodge between the wires during a sieving process.



Table 3. Comparative size analysis of a 63 micron test sieve (Endecotts Serial no. 5626574)

Certifying Laboratory	Count	Warp			Weft			X/Y mean
		Min	Max	Mean	Min	Max	Mean	
Retsch	1925	-	67.7	62.9	-	68.4	62.5	62.7
Endecotts	1600	59.8 ¹	64.4 ¹	62.1	62.0 ¹	66.2 ¹	64.1	63.1
Haver & Boecker	300	60.6	67.6	62.8	60.6	69.0	63.0	62.9
W S Tyler	300	59	67	62	59	66	62	62
Whitehouse – microsphere sieve standard								61.8
Whitehouse – near mesh method								63.5
1 Standard deviation								

The oversize microspheres not passing the sieve are removed by inverting and lightly tapping. The trapped, or 'near mesh' beads can then be removed by brushing. The variation in size of the 'near mesh' beads will reflect the variation in aperture sizes in the sieve mesh.

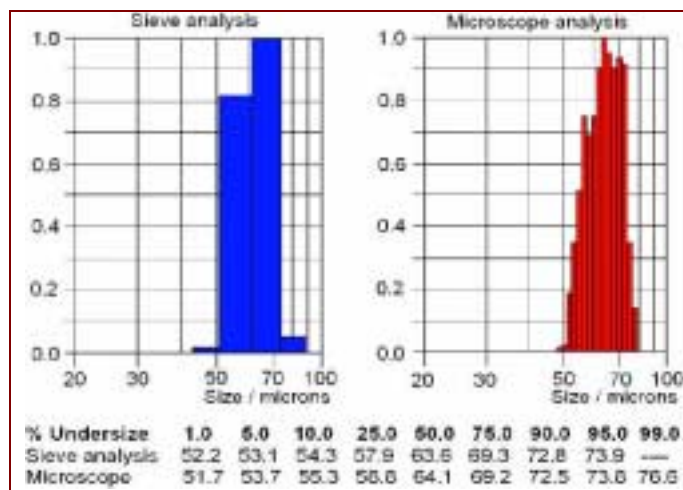
2.4 The test sieve

To ensure there was no ambiguity in the results, a single 63 micron test sieve supplied by Endecotts was circulated to all the participating laboratories (Serial number 5626574).

3 Comparative results

Full details of the ISO and ASTM methods can be found in the standards: ASTM E11 and ISO 3310 – 1:2000 respectively. To calibrate a sieve by the glass microsphere method, a single-shot vial of the calibrating microspheres was poured onto the sieve and shaken by hand or machine for 1 minute. The weight passing the sieve was then calculated and the mean aperture size read off the calibration graph. As the particle size distribution was so narrow, an uncertainty in weighing of 5% only corresponded to a size variation of 1 micron, figure 4. It is therefore not surprising that the results were very repeatable, table 2. The results of all the analyses are shown in table 3. It was very encouraging that all the international laboratories certifying the test sieve by microscopy obtained answers within about a micron of each other. Even the two glass bead methods agreed very well with the ASTM and ISO microscope methods.

Fig. 3: Results are reinforced by the higher resolution method of microscopy



4 Conclusion

Sieve analysis has been described as the 'Cinderella' of particle sizing in that it does most of the work while getting little of the credit. Furthermore, its reputation has been tainted by poor quality sieves often used by poor quality analysts. The fact remains that there are literally millions of sieves

currently in use throughout the world and it is still the cheapest way of measuring particle size by a considerable margin. It has also been shown in studies by the European Bureau of Certified Reference (BCR) that it can be the most reproducible method of particle sizing, provided stringent calibration routines are employed.

This study has shown that there is excellent agreement between all the sieve manufacturers when measuring a single sieve by prescribed international standards. Calibration by the comparatively new and convenient method using glass microspheres also gives results indistinguishable from the microscope method.

Combining the latest developments in weaving technology, sieve frame design, sieve shakers and software to calculate the results, sieve analysis could easily achieve another 5000 years of success, provided stringent analytical techniques are used to measure the sieve apertures.

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Fig. 4: Uncertainty in weighing of 5% corresponded to a size variation of only 1 micron

