False accounting

Contrary to popular belief, the humble sieve still commands a significant position in the global market. Indeed, the relationship between powder testing and processing seems as strong as ever, says Dr Graham Rideal, managing director, Whitehouse Scientific

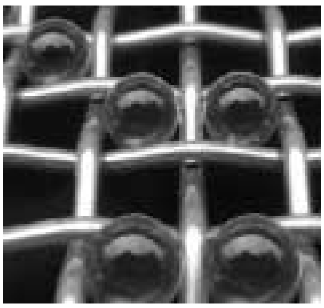
ABOVE: an Endecott powermatic for particle sizing and separation

Despite the technological developments in both particle size analysis methods and separation technologies, it is interesting that the humble sieve still commands a significant position in the global market. Rather than being in decline there are several new manufacturers, not just in traditional wire woven sieves, but also in the high precision electroformed sieves, which can be more than ten times as expensive. Indeed, the relationship between powder testing and processing seems as strong as ever. As Mark Twain once put it 'Rumours of my death have been greatly exaggerated'.

One of the main reasons for the continued success of sieving is the enormous commercial expansion in Asia, which is reported on an almost daily basis. There has also been a silent industrial revolution taking place in countries such as India. From an embryonic pharmaceutical industry, which relied almost exclusively on imports, India is now producing most of its own drugs, which for a country with a population of over 1 billion, represents a significant business by any standard. Many of the production methods use sieving for quality control, so it is hardly surprising that there has been such an increase in the sales of sieves. China, the only other country with a population in excess of 1 billion, is expected to follow the same path, albeit a few years down the road.

The emergent economies however will not accept any compromises in quality control, especially in the pharmaceutical industries. Although sieves can be calibrated by the manufacturer, the precision microscope instrumentation is far too expensive for the sieve user to buy so sieves either have to be returned or new ones purchased to maintain quality assurance.

Both of these options have their problems. In the first case, it may be impractical to return a sieve for recalibration because product testing would have to cease for several weeks while the sieve was in transit, being calibrated or simply sitting in customs. The problem with buying a new sieve is that the mesh size is almost impossible to match because of the large number of measurement parameters required in the sieve specification.



CALIBRATION REQUIREMENTS

The dimensions set by the International Standards Organisation for calibrating a sieve are:

- **Mean wire spacing at the centre of the aperture**
- **A tolerance on the mean**
- **A Standard Deviation for all the measured wire spacings**
- **A specified maximum wire spacing**
- **A specified wire thickness in the direction measured.**

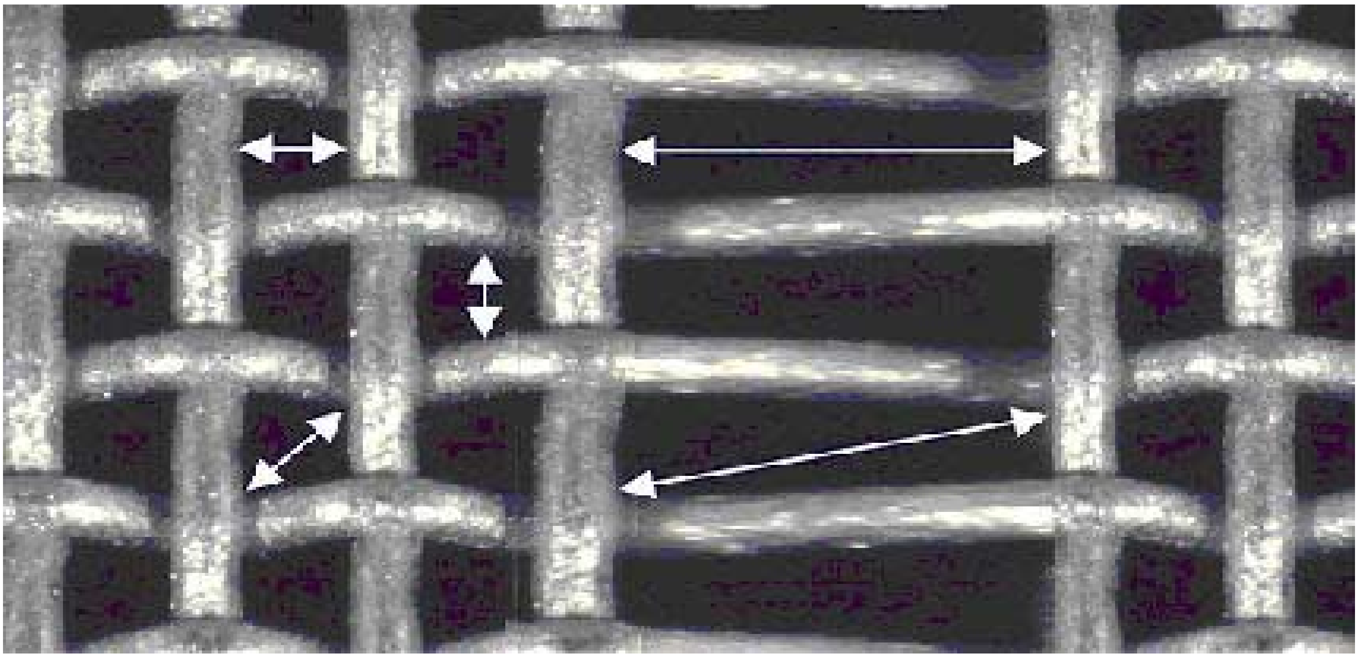
These five measurements become ten when repeated for the wires in the other direction. It can therefore be seen how difficult it is to match all ten parameters. So concerned are some pharmaceutical manufacturers than they are prepared to pay enormous premiums to scan hundreds of sieves in order to find a single match.

Needless to say, such an option is only possible for the most prestigious pharmaceutical companies and defeats the cost effectiveness of sieving technology for particle sizing.

Another area of concern is the number of microscope measurements specified per sieve, which is usually less than 1% of the apertures in the mesh. Questions therefore arise as to how representative such a small sample is of the total area.

MICROSPHERE STANDARDS

The obvious solution in calibrating a sieve is to reduce the number of measurement parameters and thus increase the probability of sieve matching. This was the approach taken by Whitehouse Scientific in



developing a unique range of sieve calibration microspheres.

The advantage of using glass microspheres is that they can be defined by a single parameter, the diameter. A sieve aperture is therefore calibrated according to an equivalent spherical diameter, a measurement that can be made traceable to an International Standard of length such as NIST (the National Institute of Standards and Technology, USA). Matching sieves is therefore much easier than the microscopic method.

An additional bonus is that the sieves do not need to be returned to the manufacturer but can be calibrated simply and quickly on site, which solves the second problem associated with intercontinental transportation.

This new approach to sieve calibration involves producing a range of narrow size distribution glass microspheres for calibrating individual test sieves from 20 – 3350 microns. The standards are certified using NIST traceable precision sieves accurate to 2 microns. As the certification method imparts a particle width dimension to the calibration microspheres, there are no particle shape implications as in the case of microscope analysis.

Because the size distributions of the standards are so narrow, only 3 or 4 electroformed sieves could be used for an analysis, so interpolation of the data could be suspect if there was not a uniform distribution of all the particle sizes across the sieve sizes used. For this reason the higher resolution technique of microscopy was used to confirm the particle size distribution and so give confidence in the interpolation of the sieving data. Only when the two sets of data were superimposable was the Electroformed sieve data used to produce a calibration graph.

SINGLE DOSE PACKAGING

In order to eliminate sampling problems, the standards are individually packaged in single dose bottles sufficient to analyse a 200 mm diameter test sieve. The weights in each bottle are calculated to analyse more than 80% of the available sieve apertures. For example, a 200 mm diameter, 63 micron test sieve has approximately 2.5 million apertures. The 1 g sample bottle contains approximately 2 million microspheres.

To calibrate a sieve, the complete contents of a single dose bottle are poured on to the sieve surface and shaken for about 1 minute. As standards are spherical they have a very high sieving rate that is independent of the shaking method. Therefore manual shaking, mechanical or electromechanical action, Air Jet and sonic sieving all give the same results.

The percentage of the microspheres passing is then used to determine the aperture size from the calibration graph on the Certificate of Analysis. Because the size distributions are so narrow, variations of 5% in the weight percentage passing corresponds to a mean aperture difference of only about 1 micron for a 63µm standard making the technique highly accurate.

SIGNIFICANT IMPACT

The introduction of these new sieve calibration standards has made a significant impact in enhancing the confidence in the results from sieve analysis. The method is simple to use giving results that are both unambiguous and conceptually easy to understand. By defining the sieve apertures as a single parameter it is now possible to successfully match test sieves and screens used for precision separation. Furthermore sieves no longer need to be returned to the manufacturer but can be calibrated on site using only an electronic balance and a calibration graph supplied.

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ABOVE: testing a faulty sieve – the ambiguity of microscope measurements.