rose-tinted glasses

Contrary to popular belief, wire woven sieves do not always have the exact aperture size specified on the label and the acceptable variation in the ISO standard may prove to be too wide for accurate results. Dr Graham Rideal, at Whitehouse Scientific explains how the use of glass microsphere sieve calibration standards is a convenient and accurate method of calibration for the end user

LEFT: single shot bottles of a sieve calibration standard being prepared on a spinning riffler.

Sieve analysis has been shown to be one of the most reliable and reproducible methods of particle size analysis, especially when highly accurate Electroformed sieves are used. Wire woven sieves can also give good results provided a prescriptive method of analysis is performed on calibrated sieves.

Unlike Electroformed sieves however, which can be manufactured to a tolerance of 1 micron, variations of up to +/- 6% may be seen in wire sieves for the same nominal sieve size. Such a variation is sometimes unacceptable for the more stringent applications, for example, in the Pharmaceutical Industry, and an exact sieve opening traceable to the International Unit of length is often essential.

Microscopy has been traditionally used to calibrate sieves but it is an expensive and time consuming method usually only available to the sieve manufacturers. It also produces two dimensions - an average warp and weft size, somewhat equivocal when a single mean aperture size is required by the user. In addition, microscopy only examines less than 1% of the total sieve area.

SIEVE CALIBRATION STANDARDS

Narrow distribution glass microspheres certified by NIST traceable electroformed sieves are available for every sieve from 20 to 3350 microns. From the percentage passing the sieve the aperture can be determined. Having narrow size distributions increases the resolution to better than one micron.

The parameter measured the method is the equivalent spherical opening of the sieve, which in practical terms is the cut point of the sieve.

Sieve calibration microspheres are much more convenient than microscopy and enable calibration to take place in the analyst's own laboratory.

SINGLE SHOT BOTTLES

One of the biggest problems of particle size analysis, irrespective of the method, is taking a representative sample. The calibration microspheres presented to the sieve must have identical particle size distributions otherwise there will be a considerable variation in the results. To overcome the sampling problem, the standards are individually packed in bottles sufficient for a single analysis, known as single shot bottles. The weights are calculated to provide sufficient microspheres to analyse at least 80% of the sieve surface, whether the sieve is 200mm or 100mm in diameter. The method used for sub-sampling is spin riffling.

Table 1: calibration of a 32 micron air jet sieve.

Test No.	Initial Sample Wt. (g)	Wt. Retained (g)	% Passing	Mean Aperture Size (microns)
1	1.02	0.20	80.4	34.0
2	1.02	0.22	78.4	33.9
3	1.04	0.21	79.8	34.0
4	1.08	0.24	77.8	33.9
5	1.01	0.23	77.7	33.9
6	1.08	0.25	76.9	33.7
7	1.05	0.24	77.1	33.7
8	0.99	0.22	77.8	33.9
9	1.02	0.22	78.4	33.9
10	1.02	0.20	80.0	34.0
Mean size 33.9 +/- 0.22 microns				

Test No.	Initial Sample Wt. (g)	Wt. Retained (g)	% Passing	Mean Aperture Size (microns)
1	0.97	0.65	33.0	59.4
2	0.97	0.67	30.9	59.3
3	0.97	0.67	30.9	59.4
4	0.96	0.67	30.2	59.4
5	0.97	0.68	30.0	59.7
6	0.98	0.69	29.6	59.5
7	0.97	0.68	30.0	59.1
8	0.97	0.67	30.9	59.3
9	0.96	0.67	30.2	59.1
10	0.97	0.68	30.0	59.4
Mean size 59.4 +/- 0.19 microns				

Table 2: calibration of a 63 micron air jet sieve.

In this method a carousel containing up to 100 bottles is rotated under a constant flow of powder. If the speed of rotation and time are calculated to give at least 100 revolutions, then the sample bottles will is contain 100 parts of the master batch, which makes it very representative.

To calibrate a sieve, the complete contents of the single shot bottle is shaken over the sieve and the aperture size calculated as above.

CALIBRATING VERY FINE SIEVES

Sieve analysis above about 53 microns is not a problem for most powders as they are relatively free flowing. However as size decreases, inter particle attraction increases resulting in poor transport through a sieve when mechanically shaken.

To effect efficient sieving, sonic energy or air jet sieving must be employed. In both these methods energy is applied to the particles rather than the sieve to produce a sharp separation. The very simple calibration procedure is outlined below:

a. Place the sieve to be calibrated on the Air Jet sieve and set the timer to 3 minutes.

b. Transfer the sieve to a balance accurate to 0.01g and tare,

c. Take a single shot bottle of the appropriate standard and empty the contents onto the sieve on the tared balance and record the initial powder weight,

d. Transfer the sieve and calibration standard back to the Air Jet sieve and start sieving. The unit will automatically stop after 3 minutes,

e. When the sieving is complete, re-weigh the sieve and record the weight of powder remaining,

f. From the weight remaining, calculate the percentage passing and use the calibration graph supplied with the Sieve Calibration Certificate to determine the mean aperture size.

The results for a 32 micrni sieve are shown in **Table 1**.

These results in **Table 1** clearly show the advantage of using a narrow size distribution calibration standard. Although the percentage passing varied from 76.9% to 80.4% this only corresponded to an aperture variation from 33.7 microns to 34 microns.

Similar excellent repeatability was seen for a 63 micron sieve, in **Table 2**.

The interesting observation from the analysis of the 63 micron sieve was that the size was actually 59.4 microns. According to the ISO specification the minimum acceptable mean aperture size is 59.3 microns so this sieve only just falls within the specification.

However, the maximum mean aperture tolerated in ISO 3310 is 66.7 microns. If the next sieve ordered by an unfortunate analyser happened to be at the other extreme of the tolerance, the results of the analysis could be quite different. This could have serious implications in certain critical industries such as pharmaceuticals.

In conclusion, contrary to some popular belief, wire woven sieves do not always have the exact aperture size specified on the label and the acceptable variation in the ISO standard may prove to be too wide for accurate results. It is therefore very important that sieves are calibrated to ensure reproducibility.

Microscopic analysis is too complicated and expensive to be employed by the end user. The use of glass microsphere sieve calibration standards has proved to be very convenient in that they can be used in any laboratory without specialist equipment. Furthermore, they are extremely accurate and the results can be traced to International Standards of length such as NIST (the National Institute of Standards and Technology, USA).

For details contact Whitehouse Scientific on tel: +44 (0) 1244 332626 or visit www.whitehousescientific.com

BELOW: the principle of sieve calibration using glass microspheres.