

Using Reference Standards in Particle Size Analysis

Given the diversity of the techniques used in particle size analysis, it is almost impossible to develop an all-embracing set of reference standards for calibration of every method. For example, in particle counters, whether employing electrical zone or optical zone methods, size channels can only be assigned accurately by using monodisperse standards.

Such standards, however, are too narrow for calibrating wire woven sieves because of the variation of aperture sizes produced in the weaving process. Particle size standards for sieve calibration should therefore be broader in distribution, but not too broad so as to limit the resolution of the method.

In the case of laser diffraction techniques, there is a requirement for both monodisperse and polydisperse standards; the former to set up the optical bench of the instrument and the latter to ensure all elements of the analysis including operator performance are rigorously tested. Polydisperse standards for laser analysis may be fully certified or just well characterised as in the case of the Malvern Quality Audit standards.

Although crushed quartz has been available as a standard for over 20 years, it has given problems in some laser diffraction particle size analysers. The source of the problem was attributed to inherent design of the early instruments, complex scattering patterns from the irregular shaped particles but much more likely, poor sample selection from the large weights supplied in the sample bottles.

More recently, the problem of particle shape has been overcome using glass microspheres and there has been for the first time, unanimity for nearly all methods. However, in the case of very small standards, for example, 3–30 microns, the incorrect application of diffraction theory can give rise to artificially generated peaks below 1 micron.

Although this is a relatively rare occurrence, manufacturers have nevertheless requested broad distribution, opaque spherical particles as the ideal reference standard.

The 'new kid on the block' in terms of particle size analysis is the new breed of Image Analysers. These combine traditional microscopy with the latest high-speed video cameras and computers to provide literally, a complete picture of the particle size and shape distributions. The images may be collected from a traditional microscope slide, a static method, or dynamically by dropping the powder in front of the video camera.

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'Single-shot' polydisperse particle size reference standards

Image analysis provides one of the highest resolution techniques in particle characterisation and as such, requires a different type of calibration standard. Not only must it have a broad distribution of sizes but ideally, internal definition or 'finger print' to quantify the high resolution of the method. Furthermore, in applications such as acicular crystals or fibre analysis, a shape standard is also required.

Historically, one of the first types of reference materials was the very narrow particle size distribution latex standards used for calibrating the Coulter Counter. While these were very useful in calibrating individual channels, they were limited in their ability to certify the dynamic range of the instruments and Polydisperse standards were then required.

One of the difficulties in using Polydisperse reference standards is obtaining a representative sub sample for analysis. Many of the problems with the early quartz samples were associated with poor sub-sampling by the operators.

A significant advance in the field of calibrating particle size instruments was the development of 'single-shot' standards where the standards are supplied in aliquots sufficient for a single analysis.

These standards are now available for almost every technique from sieving to image analysis.

In the case of sieving, there is an erroneous assumption that all the apertures in a sieve mesh are exactly the same size when in actual fact there is always a variation resulting from the weaving process and so comparisons from one sieve to another are always problematic.

A more serious concern is that some manufacturers have put the wrong sieve cloth in the frame.



Glass calibration microspheres for calibrating sieves

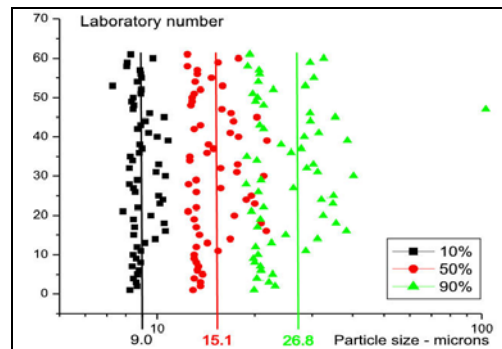
It is therefore very important that any sieve results are always supported by a certificate of calibration for each of the sieves used in the analysis.

One of the most ubiquitous techniques in particle size analysis is Laser Diffraction where accurate and high-speed results can be obtained. Although operator bias can be reduced by using single-shot polydisperse standards there can be no legislation for technicians who fail to use adequate stirring and then complain that the glass beads have disappeared, or even worse, dissolved!

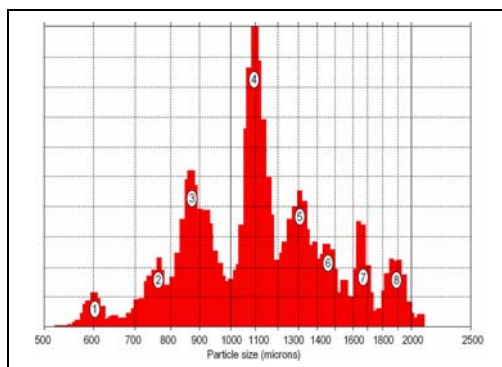
However, even in cases where operators have achieved good results with the reference standards, there is no guarantee that the results on other unknown powders will

be equally as good. This is because almost every powder has a unique set of properties and unless a pre-analysis 'titration' is performed to determine the uncertainty of measurement, there can be very large variations from lab to lab for the same sample.

A good way of evaluating the performance of a particle sizing laboratory is to ask if they are part of a Quality Audit Scheme. These schemes are very useful in developing the best working practice in particle size analysis because operators are challenged not just with reference standards but with a number of technically difficult powders to analyse.



Variation in results from a Quality Audit Scheme on a 3-30 micron Whitehouse Scientific particle size standard



A 'Multimodal' standard for checking the resolution of Image Analysers

One of the latest types of reference standard is the 'Multimodal' standard for Image Analysers. As these instruments claim very high resolution, a standard must be available to verify the claim.

In a recent case study, a long established manufacturer analysed an eight peak multimodal standard and insisted that there were only six peaks present. The same program had been used for over 10 years.

After a comprehensive investigation, it was found that a data smoothing part of the software had been erroneously left in at the development stage. Once discovered, the instrument then reported all 8 peaks in the standard.

CONCLUSION

Whatever the particle size method, the data must be supported by a calibration certificate otherwise the results are highly questionable.

From the humble sieve to the latest Image Analysers basic errors can creep in from both users and manufacturers alike and reference standards are a fundamental tool for testing methodology and accuracy.

However, even when good results are obtained with reference standards, it is no guarantee that the performance on an unknown powder will be equally as good. The ultimate test of a good particle metrology laboratory is not just the ability to replicate certificate data but whether or not they are part of a Quality Audit Scheme. You can then be sure they can handle any powder thrown at them!