Whitehouse Scientific Ltd
Whatever the particle sizing method, from sieving to the latest computerised methods...we have the perfect solution!
Whitchurch Road
Waverton
Chester CH3 7PB
UK
+44 1244 332626
WhitehouseScientific.com

Production, Certification and Use of Image Analysis Particle Size Standards

Author:
G R Rideal, J Storey
Whitehouse Scientific Ltd

ABSTRACT

Microscopy and Image Analysis is one of the highest resolution methods in particle size analysis. Any particle size reference standard produced must therefore be sufficiently challenging to adequately demonstrate the resolution of the technique. Mono-sized microspheres and even uniform, polydisperse standards do not present a sufficient challenge. A multimodal, polydisperse standard is a much more appropriate standard. This paper describes the preparation and analysis of such a standard in the range 0.5 – 2mm. An 8 peak multimodal spherical standard made from glass beads was prepared and analysed in its entirety by a NIST traceable microscopic method. The individual peaks were then fractionated by sieving and their sizes compared to those measured in the original standard. Excellent agreement was found. There was also very good agreement with an Electroformed sieving method but only at a limited number of percentile points. This low-resolution method was unable to pick out the fine detail in the standard.

1. INTRODUCTION

High-performance video cameras combined with high-speed computers have combined in recent years to provide a high-resolution method of particle size analysis. Unlike sieving or even Laser Diffraction, which are limited by the number of sieves or detection rings respectively, Image Analysis is limited only by the resolution of the camera and has been driven by the development in digital photography. With camera chips now capable of resolving over 12 megabytes of information, particles can be viewed at very high resolution. Furthermore, the images can be captured and stored at such high speeds that it is not uncommon to count over a million particles giving the technique excellent repeatability.

The earliest form of a calibration standard was a simple image of a number of opaque circles on a glass slide, which was placed between the light source and the video camera. It was hardly surprising that most Image Analysers had little difficulty in obtaining the correct answers.

Accurate Image Analysis is not just a question of pixel calibration, many other factors are involved, for example, setting the correct light levels, edge detection, dealing with touching particles or those that are intersected by the frame in the field of view. But one of the biggest problems for any particle metrologist is in taking a representative sample of what could be a few milligrams from 10's or even 100's of grams of the sample under investigation.

The Image Analysis reference standard must therefore not only challenge the performance of the instrument but the competence of the analysis in presenting the sample to the analyser.

In order to measure the effective dynamic range of the Image Analyser, a wide particle size distribution standard is to be preferred but a simple log normal distribution is not a sufficient challenge because algorithms in computer software can be applied to 'fit' the data to a predetermined model.

The behavioural properties of a powder is often governed by factors other than simple particle size distribution, for example subtle combinations of different particle sizes and shapes. It in this area that Image Analysis excels. This paper describes the preparation and analysis of a multimodal, polydisperse glass standard in the size range 0.5 – 2mm. The complete sample was initially measured to determine the size of the peaks. It was then fractionated by sieving into the component parts and re-analysed to confirm the position of the peaks.

2. PREPARING A MULTIMODAL, POLYDISPERSE STANDARD

The glass beads component parts for the multimodal standard were produced by sieving from bulk samples and shape sorting to remove the non-spherical particles. The nominal fractions produced were 500–600, 600–710, 710–850, 850–1000, 1000–1180 microns etc. (an ISO sieve progression). As far as it was possible, only non-overlapping fractions were recombined so that the final sample had individual well-defined peaks.

The master batch was spin riffled into 10g sub samples on a 100 stage spinning riffler. The complete 10g sample was used for Electroformed sieve analysis, but the 10g samples were further riffled into 1g sub-samples for analysis on the microscope.

3. FRACTIONATING THE INDIVIDUAL PEAKS

5kg of the standard was taken and split into the component parts on a 450mm diameter sieve shaker. The fractions were then subdivided into 1g bottles for Image Analysis.

4. MICROSCOPY AND IMAGE ANALYSIS

The pixels on the camera were first measured in the X and Y direction and referenced to the square grid from a National Physical Laboratory particle reference graticule. An aspect ratio correction was applied to ensure that there was no image distortion during analysis (differences of up to 7% have been seen for some cameras). The microscope could then be calibrated at different magnifications.

A Navitar zoom microscope with indents at fixed magnifications was used with transmitted light. In this work the magnification was set to give a resolution of 0.0170 mm per pixel. The Miles-Lantuejoul correction factor was applied to compensate for particles cut by the frame in the microscope field of view. Results were converted to a volume basis so they could be compared with other primary methods of analysis eg. sieve analysis.

The 1g sub-samples of the beads to be analysed were scattered on a 5 x 20mm adhesive transparent tape tensioned across a large piece of glass on the microscope stage. This method ensured that the beads did not roll away and even gave the opportunity of physically separating any particle clusters using a small probe. By systematically traversing this large slide, it was possible to analyse all the particles present in the 1g sub-sample bottles.

5. ELECTROFORMED SIEVE ANALYSIS

Electroformed sieves in transparent 90mm diameter frames were used for the analysis. The aperture sizes were: 500, 600, 710, 850, 1000, 1180, 1400, 1700 and 2000 microns and were NIST certified by microscopy at a magnification corresponding to a field of view containing 4 apertures to give the highest accuracy.

Being transparent, it was easy to see when the beads stopped passing the sieves and the end point had been reached. A 5 minute period on the sieve shaker was used.

6. RESULTS

6.1 Fine detail of the 500-2000μm Image Analysis Standard

The detailed results for the 3 microscope analyses of the 500-2000 micron multimodal standard is shown in figure 1. No smoothing or curve fitting was employed and the sizes were grouped into 64 channels. The 8 component peaks can be clearly seen.

6.2 Analysis of the fractionated peaks

The Image Analysis of the 1g bottles of the fractionated components is shown in detail in table 1.

It can be seen that, within experimental error, the particle sizes of the individual (separated) peaks are identical to those measured collectively on the complete standard.

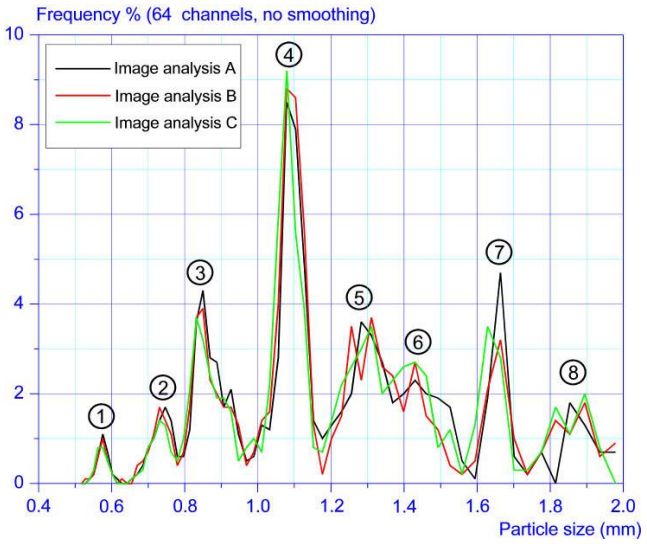

Fig 1. Microscope Analysis of a 500-2000μm IA Standard

Table 1. Comparison of the fractionated peaks sizes with those found on the original standard

Fraction	Percentile			Count	Mode	Average Mode (μm)	Average Mode (μm)	%
I.D[1]	5%	50%	95%			(fraction)	(standard)	difference
#1A	0.562	0.596	0.622	779	0.599	0.600	0.576	4.1
#1B	0.575	0.604	0.639	638	0.601			
#1C	0.559	0.601	0.631	618	0.600			
#1D	0.559	0.598	0.622	652	0.599			
#1E	0.566	0.600	0.626	638	0.602			
#2A	0.685	0.748	0.779	570	0.753	0.756	0.736	2.6
#2B	0.690	0.743	0.778	649	0.764			
#2C	0.628	0.739	0.774	614	0.759			
#2D	0.680	0.741	0.776	647	0.752			
#2E	0.688	0.742	0.776	586	0.750			
#3A	0.786	0.863	0.955	634	0.854	0.857	0.844	1.5
#3B	0.785	0.869	0.954	605	0.862			
#3C	0.787	0.874	0.962	620	0.874			
#3D	0.779	0.861	0.953	1232	0.847			
#3E	0.777	0.859	0.945	611	0.850			
#4A	1.020	1.101	1.190	675	1.099	1.108	1.079	2.6
#4B	1.023	1.103	1.167	607	1.119			
#4C	1.017	1.102	1.167	619	1.116			
#4D	1.020	1.100	1.170	1100	1.100			
#4E	1.022	1.097	1.154	578	1.108			
#5A	1.146	1.293	1.364	540	1.280	1.295	1.302	0.5
#5B	1.222	1.300	1.369	587	1.314			
#5C	1.106	1.289	1.381	591	1.320			
#5D	1.106	1.284	1.366	559	1.274			
#5E	1.092	1.284	1.355	571	1.285			
#6A	1.374	1.450	1.565	578	1.468	1.447	1.430	1.2
#6B	1.365	1.447	1.563	558	1.446			
#6C	1.340	1.445	1.551	587	1.437			
#6D	1.329	1.428	1.546	510	1.461			
#6E	1.329	1.425	1.528	595	1.425			
#7A	1.609	1.655	1.707	515	1.650	1.647	1.652	0.3
#7B	1.603	1.654	1.714	522	1.651			
#7C	1.599	1.657	1.719	520	1.670			
#7D	1.542	1.636	1.702	515	1.636			
#7E	1.570	1.638	1.691	537	1.629			

#8A	1.797	1.867	1.993	605	1.866	1.865	1.881	0.9
#8B	1.796	1.882	1.987	528	1.863			
#8C	1.807	1.883	1.987	533	1.887			
#8D	1.778	1.858	1.960	519	1.840			
#8E	1.775	1.860	1.902	565	1.870			
1. Fractions 1 – 8 in figure 1								

6.3 Comparison of Image Analysis Results with Electroformed Sieve Analysis

Electroformed sieve analysis is an example of a low-resolution method of particle size analysis because it is limited in resolution by the size of the sieves available. However, in several round-robin measurements it consistently outperforms many other so-called high-tech methods. It therefore makes an excellent comparative particle sizing method for Image Analysis.

The cumulative percent undersize data at fixed percentiles is shown in table 2.

Table 2. Comparison of Microscopy and Sieve Analysis for a 500–2000μm Standard

Percentile	**5**	**10**	**25**	**50**	**75**	**90**	**95**
Microscopy[1]	716	781	901	1102	1343	1658	1803
(Uncertainty)	1	4	16	0	14	10	37
Sieving[2]	724	796	936	1098	1335	1618	1757
(Uncertainty)	5	10	9	11	32	40	57

1. Total count 10,220 (3 x 5g) - volume average

2. Sieving 5 x 10g - weight average

The results from the two techniques are inseparable, which is well illustrated in figure 2.

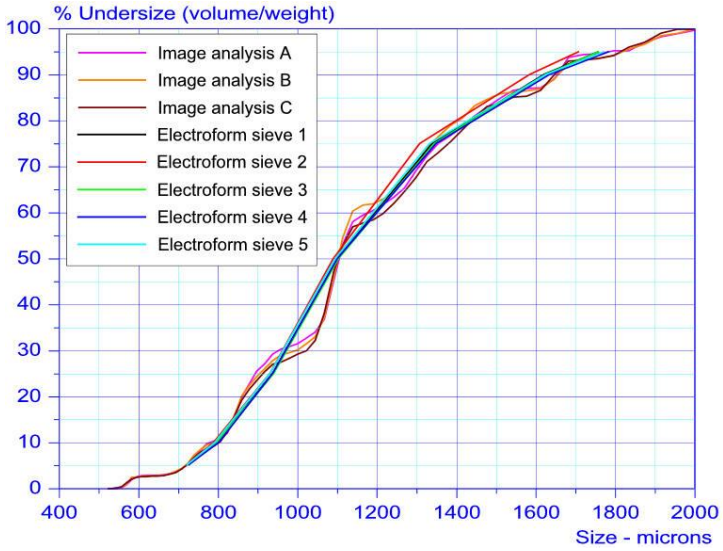

Fig 2. Comparison of Image Analysis and Sieve data for a 500–2000μm IA Standard

7. CONCLUSIONS

A highly challenging particle size reference standard has been prepared for Image Analysis that fully exploits the significant advances that have been made in the technique in recent years. The multimodal, polydisperse standard comprising of 8 individual peaks was designed to exploit the high-resolution advantage of the method. The accuracy of the assignment of the peaks using a simple laboratory microscope was confirmed by separating the peaks by sieving and analysing them individually. Excellent agreement was found. The complete 500–2000 micron standard was also measured by the highly accurate, but lower resolution method of Electroformed sieving and again, the results were super-imposable.

The new Image Analysis standard therefore represents a good challenge for the latest instruments and, because it is in the form of a dry powder, can be used in fully automated systems. The 200g bottles contain over 100,000 beads so the results should be statistically very robust.