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PRECISION CALIBRATION MICROSPHERES FOR THE HOSOKAWA MICRON ALPINE AIR JET SIEVE

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Summary

This paper describes the preparation and use of precision glass microspheres for calibrating Hosokawa Alpine Air Jet Sieves from 20 to 1000 microns. To test the accuracy and repeatability of the method, four sieves from 32 microns to 500 microns were calibrated by a method that gives traceability to International Standards.

Unlike microscopy which only examines a small fraction of the sieve apertures, the method described in this report examines over 80% of the sieve surface in about 3 minutes with a measurement uncertainty of only 1 - 2% (less than 1 micron in most cases).

Key Words: Sieve analysis, Calibration, Standards, Microspheres, Air Jet

1. Introduction

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[1,2]. 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 specified.

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 are more efficient in covering the sieve surface and have been produced in the past as wide distribution crushed quartz[3] or glass beads[4]. In the former, the irregular shape of the powder leads to confusion on the exact parameter required to pass the sieve, while in the latter, although the beads are mostly spherical, they are certified by microscopy not sieving so any deviation from the perfect sphere will give rise to inaccuracies.

In this work narrow distribution glass microspheres have been produced for every sieve from 20 to 1000 microns and calibrated by the highly accurate method of electroformed sieve analysis. Having narrow size distributions increases the resolution when converting the percentage passing the sieve into an aperture size. The parameter measured by calibration microspheres is the minimum mean aperture size, irrespective of whether it is the warp or weft dimension, as it is this dimension that restrains spherical particles.

The Air Jet sieve is well known both for its excellent reproducibility and its capability of dry sieving down to 20 microns[5] (10 microns when using Electroformed sieves). This paper examines the repeatability of the Air Jet sieve with respect to the sieve calibration microspheres.

2. Experimental

2.1. Preparation of Sieve Calibration Standards

The objective of a high precision sieve calibration standard is to provide a distribution of spherical beads which peaks at the nominal aperture of the sieve to be calibrated whilst having very little sample above or below the sieve either side of the standard sieve series. Thus for a 63 micron sieve calibration standard, about of 90% by weight of the powder should be between 53 and 75 microns with approximately 50% remaining on the 63 micron sieve.

A Production Sonic Siever from Gilson[6], see figure 1, was used to prepare the standards because it gives exceptionally sharp cuts due to the intense sonic energy used in fluidising the powders. In addition to its efficiency of separation, it also has high throughputs (up to 100kg/hr from an 8 inch or 200mm square sieve) and can dry sieve down to 20 microns.

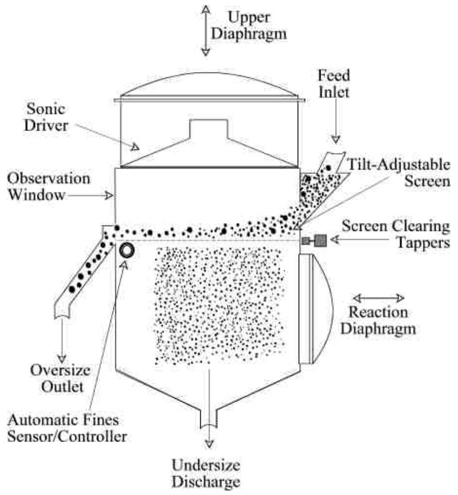

figure 1: A Gilson production Sonic Siever

In the preparation of the 63 micron standard, broad distribution glass microspheres were first sieved at 75 microns and the undersize returned over a 53 microns sieve to eliminate the fines. As the residence times were only 8-10 seconds, 3 passes were required to ensure all the fines had been removed.

2.2. Sample subdivision

From preliminary tests on 200mm sieves it was found that only 0.8 - 3g of microspheres were required to calibrate sieves from 20 to 1000 microns respectively. 8 to 30kg master batches were therefore prepared and spin riffled into 100 sub-samples using a specially constructed 100 stage spinning riffler. Each 100th sample was then further subdivided on the riffler to produce 10,000 bottles of 0.8 - 3g calibration standards. The accuracy of the riffler was confirmed by the European Community Bureau of Certified Reference (BCR) and used for the sub-division of its own general purpose reference standards[7].

The complete range of standards produced is shown in table 1.

Sieve Size (μm)	Nominal Sample Range (μm)	Nominal Sample Weight (g)
1000	850-1180	3.0
850	710-1000	2.5
710	600-850	2.5
600	500-710	2.5
500	425-600	2.5
425	355-500	2.5
355	300-425	2.5
300	250-355	2.5
250	212-300	2.5
212	180-250	1.5
180	150-212	1.5
150	125-180	1.5
125	106-150	1.0
106	90-125	1.0
90	75-106	1.0
75	63-90	1.0
63	53-75	1.0
53	45-63	1.0
45	38-53	1.0
38	32-45	1.0
32	25-38	1.0
25	20-32	0.8
20	15-25	0.8

Table 1: Weight and Size Range of the Air Jet Sieve Calibration Standards

2.3. Calibration of the sieve standards

All the sieve calibration standards from 1000 microns down to 20 microns were certified using electroformed sieves. These highly accurate sieves have perfectly square apertures accurate to 2 microns and are mounted in 75mm clear plastic frames so that the sieving process can be monitored. The traceability to the International Unit of Length was via the NPL microscope reference graticule[8]. For sizes above 53 microns, an electromagnetic sieve shaker was used while the finer grades were measured on the laboratory version of the Gilson Sonic Sifter (model GA-6). 5 measurements were made for each grade and the repeatability was better than 1% in most cases.

For the 63 micron standard the standard deviation was never greater than 0.16 microns, exceptional by any method of particle size analysis, see figure 2.

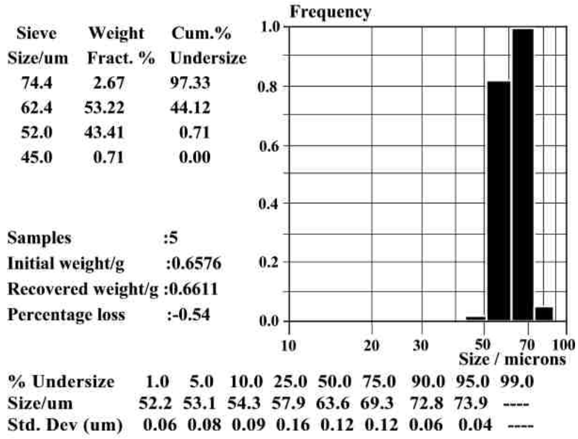

figure 2: Analysis of a 63µm sieve standard by electroformed sieves – average of 5 tests

2.4. The sieving action of the Air Jet Sieve

In the sieving process a partial vacuum is generated in a chamber below the sieve which causes the particles being sieved to be sucked onto the surface of the sieve mesh. (figure 3) The air removed is replaced through a rotating nozzle forming a circulating curtain of air, or jet, focused beneath the particles adhering to the sieve surface. As a result, the particles are blasted off the surface and are immediately sucked back once the influence of the air jet has passed. In the case of larger particles, for example, above 100 microns, the sample movement is simply up and down as the rotor sweeps by, but for smaller sizes, powerful secondary cyclonic air currents are developed generating intense turbulence over the sieve surface. This produces a highly efficient separation of the finest particles enabling the minimum size to be extended well below that of traditional dry sieving methods.

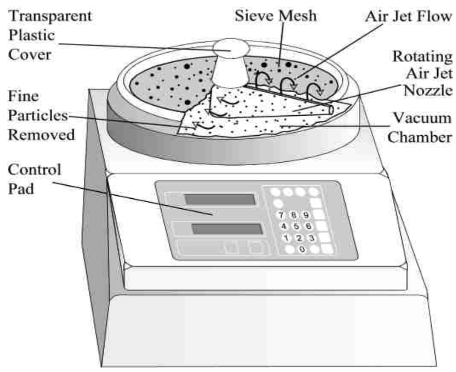

figure 3: The Hosokawa Alpine Air Jet Sieve

2.5 Sieve Calibration Method

10 repeat analyses were performed on each 32, 63, 106 and 500 micron sieve using the following calibration procedure:

a. Place the sieve to be calibrated on the Air Jet sieve,
   Set the timer to 3 minutes, start the unit and adjust the vacuum to 2000 - 2200 Pa,
b. Transfer the sieve to a balance accurate to 0.01g and tare,
c. Take the first 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. In the meantime carefully brush off any particles of the standard that have fallen through the sieve onto the balance,
f. When the sieving is complete, re-weigh the sieve on the tared balance and record the weight of powder remaining,
g. 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, see figure 4,
h. Carefully clean the sieve, ideally until the reading on the tared balance returns to zero.

Test No.	Initial Sample Wt. (g)	Wt. Retained on Sieve (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 with a confidence level of not less than 95%				

Table 2: Calibration of a 32µm Air Jet Sieve - a repeatability study

3. Calibration Results

3.1. 32 micron test sieve

Using the procedure described above and the calibration graph supplied with each certificate, figure 4, the mean aperture size of the 32 micron sieve was found to be 33.9+/- 0.22 microns, table 2.

These results clearly show the advantage of using a narrow size distribution calibration standard. The maximum variation in the percent passing was 3.5% which corresponds to a mean aperture size difference of only 0.3 microns.

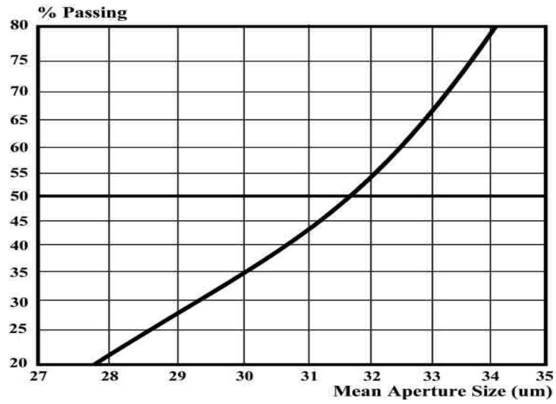

figure 4: Analysis of a 63μm sieve standard by electroformed sieves – average of 5 tests

3.2. 63 micron test sieve

The results of 10 calibrations of a 63 micron Air Jet sieve is shown in table 3.

Test No.	Initial Sample Wt. (g)	Wt. Retained on Sieve (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 59.2 +/- 0.19 microns with a confidence level of not less than 95%				

Table 3. Calibration of a 63 micron Air Jet sieve - a reproducibility study

The mean aperture size was found to be 59.2+/-0.22 microns or 6.03% below the nominal size which is just about within specification.

The result on the 63 micron sieve using the Air Jet method was almost identical to that obtained by both manually shaking the sieve or using an Electromagnetic sieve shaker showing that the accuracy of the technique is independent of the method of shaking[9].

3.3. 106 micron test sieve

The results of 10 calibrations of a 106 micron Air Jet sieve shown in table 4 above, re-enforces the repeatability of the precision microsphere method giving a mean aperture size of 102 +/-1.23 microns.

Test No.	Initial Sample Wt. (g)	Wt. Retained on Sieve (g)	% Passing	Mean Aperture Size (microns)
1	0.95	0.49	48.4	103.5
2	0.94	0.52	44.7	102.0
3	0.97	0.53	45.4	122.2
4	0.99	0.57	42.4	101.0
5	0.99	0.55	44.4	101.8
6	0.96	0.54	43.8	101.6
7	0.95	0.53	44.2	101.8
8	0.95	0.53	44.2	101.8
9	0.97	0.53	45.3	102.2
10	0.94	0.52	44.7	102.0
Mean size 102.0 +/- 1.23μm with a confidence level of not less than 95%				

Table 4: Calibration of a 106μm Air Jet Sieve - a reproducibility study

3.4. 500 micron test sieve

Test No.	Initial Samples Wt. (g)	Wt. Retained on Sieve (g)	% Passing	Mean Aperture Size (microns)
1	2.43	1.20	50.6	495
2	2.44	1.23	48.6	493
3	2.42	1.21	50.0	493
4	2.42	1.20	50.4	495
5	2.43	1.22	49.8	493
6	2.45	1.23	49.8	493
7	2.37	1.19	49.8	493
8	2.43	1.21	50.2	494
9	2.39	1.17	51.0	495
10	2.46	1.21	50.8	495
Mean size 494 +/- 1 micron with a confidence level of not less than 95%				

Table 5: Calibration of a 500 micron Air Jet Sieve - a reproducibility study

The results from the 500 micron sieve are shown in Table 5. In this case the absolute variation seen was only 493 - 495 microns - an uncertainty of only 1 micron.

4. Conclusions

This work confirms the excellent repeatability that can be achieved in sieve analysis using the Hosokawa Alpine Air Jet Sieve. It also shows that precision microsphere standards are an extremely fast and accurate method of calibrating sieves; a typical analysis taking just 3 minutes to calibrate to a traceable International units of length. The results were all the more encouraging because as little as 1 gram of standard was required to give a repeatability of below 1% with a confidence level of not less than 95%. An additional advantage of the reference standard calibration method is that over 80% of the sieve surface is analysed compared with a microscopic measurement which takes 10 times longer to analyse less than 1% of the apertures[9].

Furthermore, the results are expressed as a single parameter, the equivalent spherical or minimum mean aperture size, rather than the equivocal double measurements of mean warp and weft determined microscopically. Because the results are so repeatable, only a single analysis by the particle size analyst is required provided the guidelines set out in this paper are followed.

5. Acknowledgements

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7. References

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