

SIEVE CALIBRATION - A NEW SIMPLE BUT HIGH PRECISION APPROACH

G R Rideal, J Storey, T R Morris, Whitehouse Scientific Ltd, Chester, U.K

Abstract

This paper describes the preparation, measurement and use of microspheres for calibrating individual test sieves. Using a 63 μm sieve as an example, 2.5 million apertures, or 80% of the surface is examined in under 2 minutes. Because of the narrowness of the size distribution, a 5% difference in percent passing only results in a mean aperture difference of 1 μm , indeed, the measurement uncertainty for all 24 tests performed was only 0.7 μm . The calibration is independent of the method of shaking and can be used for most sieves.

1. Introduction

Electroformed Sieve analysis has been shown to be one of the most reliable and reproducible methods of particle size analysis^{1,2}. However, significant variations from the nominal sieve aperture may occur in the less accurate woven wire sieves. For example, the largest permitted aperture in a 63 micron (μm) sieve is 89 μm ³. In practice such extremes are rarely seen but they could have a profound effect on the analytical results. Furthermore, there have been occasions where human error has resulted in sieve frames being wrongly labelled. Sieve calibration is therefore essential for precise measurement. Microscopy has been successfully used to calibrate wire woven sieves but, for every day use, it has several disadvantages:

- dedicated image analysis is specialised and expensive,
- the mean warp and weft measured are equivocal and only indirectly related to sieving performance,
- the method typically examines less than 1% of the total number of apertures on a sieve surface,
- for good statistics, the high aperture count required makes the method very time consuming,
- the geometry of the weave results in the path of a particle through the sieve being different from the optical path of the transmitted light used for analysis.

An alternative method to microscopy is the use of reference standards^{4,5} which analyse a much larger area of the sieves. The BCR quartz standards are non-spherical so the results are shape dependent while the spherical NIST standards are certified by microscopy rather than directly by electroformed sieving so any non-sphericity can lead to significant differences. In both cases the size distributions are very wide. For maximum accuracy, sieving standards should be certified by electroformed sieves, be spherical and narrow in particle size distribution for the following reasons:

- the apertures can be described by a single parameter,
- narrow distributions give higher resolution results,
- spherical particles have high sieving rates,
- spherical particles enable more accurate comparisons to be made with other methods such as microscopy.

2. Preparation of Sieve Calibration Standards

The objective of a single 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 μm sieve calibration standard, about of 90% by weight of the powder should be between 53 and 75 μm with approximately 50% remaining on the 63 μm sieve.

A Production Sonic Siever from Gilson (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 μm .

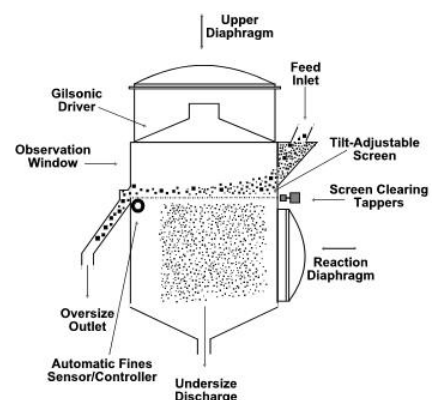


Fig. 1: A Gilson production sonic siever

For the preparation of the 63 μ m standard, broad distribution glass microspheres were first sieved at 75 μ m and the undersize returned over a 53 μ m 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.

3. Certification of a 63 μ m Reference Standard

3.1. Sample Subdivision

A 10kg batch of the 63 μ m reference standard was prepared and spin riffled⁶ into 10 sub-samples. Each 1kg sample was then further subdivided on a 100 stage spinning riffler⁷ to produce 1000 x 10g samples. 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 standards⁸.

A final single 10g sample was further subdivided by spin riffler into 16 x 0.6g sub-samples for analysis by electroformed sieve analysis.

3.2. Calibration of the Electroformed Sieves

The 75mm diameter electroformed sieves were supplied by the Gilson Company⁹ using Buckbee Mears¹⁰ unsupported membranes in transparent plastic frames. They were calibrated by the Shapersizer Image Analyser¹¹ referenced to the International unit of length by the National Physical Laboratory stage graticule¹². The microscope resolution (pixel size) was set to about 0.5 μ m which corresponded to 6 apertures per field of view and then 60 apertures were examined. The minimum mid point dimension was measured as it is this dimension that retains a spherical particle.

The high precision of the 63 μ m electroformed sieve is shown in figure 2. Although the size range measured was only about +/- 1 μ m, the actual aperture precision is even greater as the microscope resolution accounts for +/-0.5 μ m.

3.3. Electroformed Sieve Analysis

A sieve stack from 45 - 75 μ m was assembled on a Fritsch A21 sieve shaker¹³ and 5 of the 0.6g sub-samples run for 3 minutes. The results are shown in figure 3. When plotted on a cumulative basis, the 5 analyses could not be separated and differences could only be seen when each result was expressed as a percentage deviation from the mean.

The result of each analysis, interpolated at fixed percentile values is shown in figure 4.

The mean standard deviation of only +/-0.17% highlights the excellent repeatability of the electroformed sieve analysis and illustrates the power of this method as a primary or absolute particle size measurement.

3.4. Image Analysis of the 63 μ m Sieve Standard

Because only 4 sieves were used in the analysis, a bimodal distribution would not easily be detected and so introduce potential errors in the interpolations to be performed. The sieve analysis was therefore backed up by the higher resolution method of microscopy and image analysis.

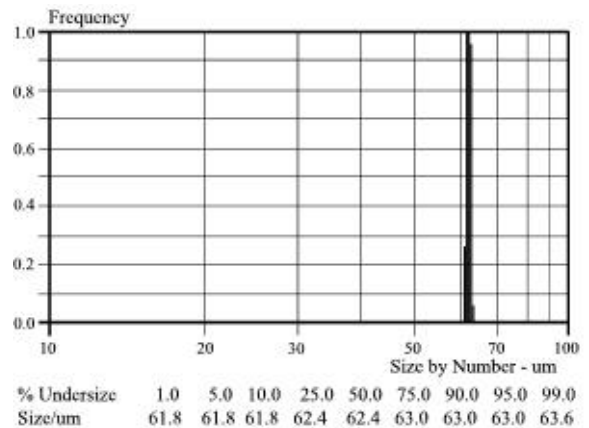


Fig. 2: Microscope calibration of a 63 μ m electroform sieve

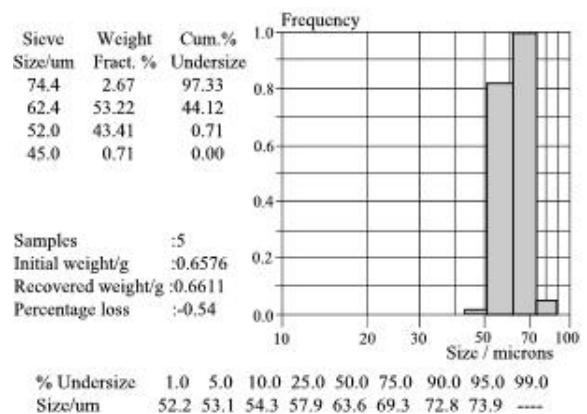


Fig. 3: Analysis of a 63 μ m sieve standard by electroformed sieves – Average of 5 tests

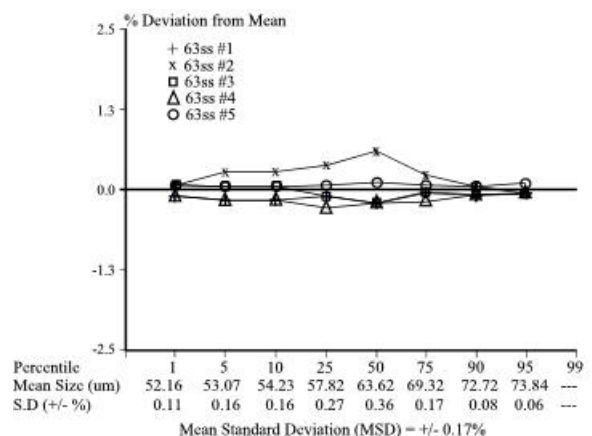


Fig. 4: Electroformed sieve analysis of a 63 μ m sieve calibration standard repeatability experiment

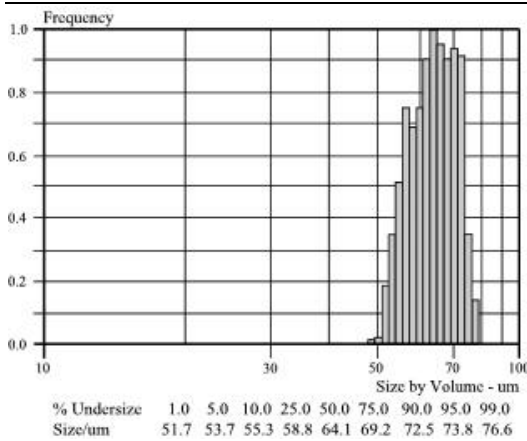


Fig. 5: Microscope analysis of a 63µm sieve calibration standard – by volume experiment

The Shapersizer analysis, converted to a volume basis for comparison, is shown in figure 5.

The single uniform peak has an almost identical distribution the electroformed sieve analysis data of figure 3. The results of electroformed sieve analysis and microscope analysis were then compares at fixed percentiles - figure 6.

The close agreement between the two methods confirms the advantage of using microspheres for the sieve calibration standards as much greater differences would be expected for non-spherical or irregular particles. Furthermore, the uniformity of the distribution gives confidence in the interpolation of the electroformed sieve analysis results. Size data from 20 to 80% in 5% intervals were then used to produce a log size versus probability sieve calibration graph for the Test Certificate - see Figure 7

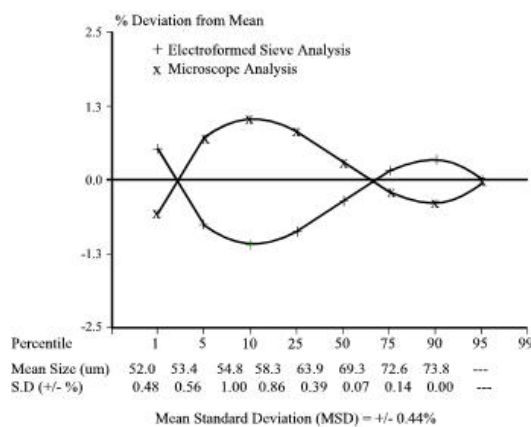


Fig. 6: Comparison of electroformed sieve and microscope data

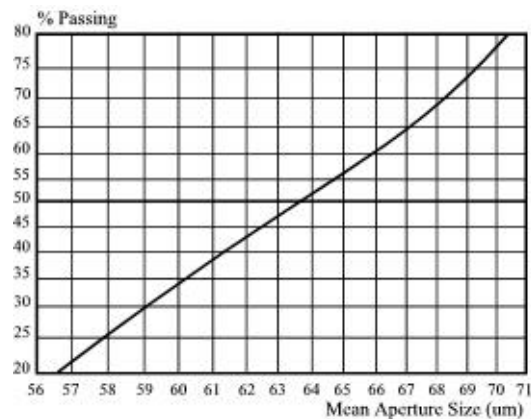


Fig. 7: Sieve calibration graph for a 63µm sieve standard

For a true Gaussian distribution this should be a straight line, but in practise, there is usually a slight deviation. The advantage of the narrow size distributions for the sieve standards can be clearly seen in that a difference of 5% in percentage passing only results in a mean aperture difference of about 1µm, making the reference microsphere method a high resolution technique of calibrating sieves.

4. Calibrating a 63µm Wire Woven Test Sieve

4.1. Microsphere Reference Standard Method

The first experiment was designed to examine the effect of sample weight and shaking method on the mean aperture size. The sieve to be calibrated was a 63µm Hozokawa Micron Air Jet Sieve¹⁴.

The sieve was weighed, clamped on the Fritsch sieve shaker and 10g of the sieve calibration standard shaken for 5 minutes. Although there was evidence of sieve blinding in the first test, the efficiency was not impaired as halving the weight did not affect the result, even though the sieving time was also reduced to 2 minutes. See Table 1. Reducing the weight to 1g and using the Air Jet sieve did not significantly change of the mean aperture size so the method of mechanical shaking did not appear to affect the result.

Table 1: 63µm wire sieve calibration results - effect of different sieve shaking methods.

Test No.	Sample Wt. (g)	Conditions	% Passing	Mean Aperture (µm)
1	9.94	5 mins Fritsch	31.8	59.4
2	4.91	2 mins Fritsch	32.0	59.4
3	0.97	2 mins Air Jet	33.0	59.5
4	2.18	2 mins hand	33.5	59.7
5	1.10	2 mins hand, 1min Fritsch	30.0	59.0
Average Mean Aperture = 59.4µm S.D. = 0.25µm (0.42%)				

After each test, the sieve was cleaned by inverting and brushing the underside with a paint brush until no visible particles remained and the tare weight returned to zero. The mechanical methods were then compared to hand sieving. 2g of powder was shaken for the same time of 2 minutes using a large swirling or oscillatory action to ensure total coverage of the mesh surface. On completion, the frame was tapped a few times to remove near mesh particles before measuring the percent passing. Again, there was no significant difference in the mean aperture. Finally, 1g of powder was shaken by a combination of mechanical and hand shaking. There was, once again no significant difference on the calculated mean aperture. As hand shaking seemed to be so effective, the next series of experiments investigated the effect of shaking time on the result. In this case, a 1.3g riffled sample was shaken for times varying from 15 to 75 seconds. No further changes were seen after 60 seconds; the final mean aperture being very close to the first series of experiments - Table 2.

Table 2: 63 μ m wire sieve calibration - hand shaking 1.3g riffled samples.

Time (sec)	% Passing	Mean Aperture (μ m)
15	30.0	59.0
30	30.8	59.1
45	31.5	59.4
60	32.3	59.5
75	32.3	59.5
Final Mean Aperture = 59.5 μ m SD = 0.19 μ m		

Having determined the end point of the hand sieving method, the next series of experiments examined the repeatability of the calibration process. 5 of the 1.3g riffled samples were hand shaken for 75 seconds. Care was again taken to ensure that, in the shaking process, the entire surface of the mesh was covered by the calibrating microspheres. The result was very surprising, and most encouraging in that the hand shaking method was extremely reproducible - see table 3.

Table 3: 63 μ m sieve calibration results for riffled samples - hand shaking 75 seconds.

Sample Wt. (g)	% Passing	Mean Aperture (μ m)
1.31	32.8	59.6
1.31	32.8	59.6
1.31	32.8	59.6
1.31	32.8	59.6
1.30	33.8	59.8
Average Mean Aperture = 59.6 μ m SD = 0.08 μ m (0.13%)		

The final results of table 3 were virtually identical to the first two series and clearly show that calibrating sieves by reference microspheres is fast, highly accurate and repeatable, especially when using riffled samples. Riffing is very important for obtaining a representative sample of a broad range distribution powder⁶, but in the case of these narrow size distribution single sieve standards, riffing may not be so important.

The final series of experiments examined the repeatability obtained by pouring out successive 1 gram samples of powder from the 10g bottle. Before each sample was taken, the bottle was thoroughly shaken by hand for about 15 seconds. The results for the 10 analyses is shown in table 4. Although of standard deviation is not as good as the riffled samples, the mean aperture is once again indistinguishable.

Table 4: 63 μ m wire sieve calibration - repeatability within 10g bottle - 1g samples poured out after shaking bottle.

Sieve Weight (g)	Sample Weight (g)	Wt After Sieving (g)	% Passing	Mean Aperture (μ m)
282.94	1.00	283.63	31.0	59.1
282.94	1.00	283.63	31.0	59.1
282.94	1.00	283.62	32.0	59.4
282.94	1.00	283.61	33.0	59.7
282.94	1.00	283.61	33.0	59.7
282.94	1.00	283.64	30.0	59.0
282.94	1.00	283.64	30.0	59.0
282.94	1.00	283.62	32.0	59.4
282.94	1.00	283.63	31.0	59.1
282.95	0.95	283.57	34.7	60.1
Average Mean Aperture = 59.4 μ m S.D. = 0.35 μ m (0.59%)				

4.2. Near Mesh Method of Calibration

An alternative technique to the microsphere reference standard method is the near mesh method of calibrating sieves. Here, after the sieving process is complete and the oversize fraction is removed, the particles loosely held in the sieve wire are dislodged by gently tapping the sieve frame and those held firmly in the mesh (the near mesh particles) are then brushed out for analysis by microscope. The near mesh particles should be close to the range of aperture sizes in the sieve cloth although in practise they are usually a little larger for several reasons.

Firstly they tend to be trapped below the maximum diameter in order to wedge in the weave, secondly, slightly out of shape beads have a larger microscope size (equivalent spherical diameter) than the sieve diameter, and thirdly any wire movement in the sieve could create a larger effective aperture.

The Shapersizer analysis of the near mesh microspheres, figure 8, confirms a larger mean aperture size than the sieving method - 63.5 μm compared to 59.6 μm , however, without an uncertainty analysis on the near mesh method, it is not clear whether this 3.9 μm (6.5%) difference is significant.

4.3. Calibration by Microscope

The 63 μm sieve was also analysed by microscope using the sieve calibration facility of the Shapersizer. By measuring the maximum and minimum mid point dimension of each aperture, both the size and shape can be analysed. The results of measuring 200 apertures evenly selected over the surface of the 200mm diameter sieve is shown in figure 9.

The median size of 60.5 μm is in very close agreement with the 59.6 μm obtained from the microsphere calibration method - Table 3. The shape distribution of the aperture dimensions is shown in figure 10. The shape factor (SF), maximum to minimum ratio, can be used to convert the mean measured size from minimum to maximum. Thus multiplying the mean minimum size of 60.5 μm by 1.03 gives a mean maximum aperture size 62.3 μm . While the maximum dimension may be relevant for irregular particles it does not affect the analysis of the spherical particles used in this work.

5. Discussion and Conclusions

Ideally the aperture size of a calibrated sieve should be expressed as a single dimension that is unambiguous, highly accurate, repeatable, and traceable to the International unit of length. The calibration should also analyse a high percentage of the sieve surface and, from the analyst's perspective, be a simple and speedy operation. The near mesh method is difficult to perform, time consuming and not traceable because it is uncertain how much extra width over the diameter is necessary for the bead to wedge in the mesh. It is also shape dependent.

Direct measurement by microscopy is ambiguous in that it produces 2 dimensions. It also only analyses a very small

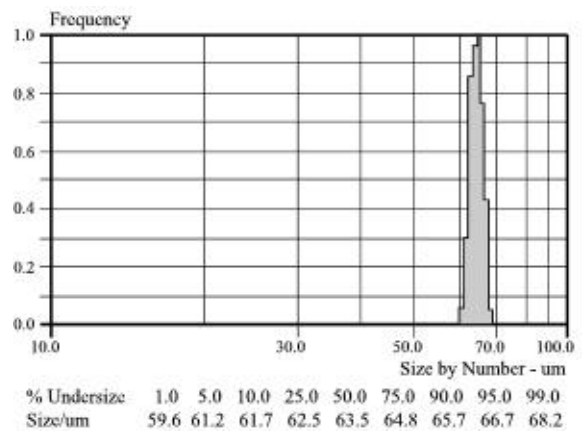


Fig. 8: Analysis of 63 μm near mesh microspheres from a wire woven sieve

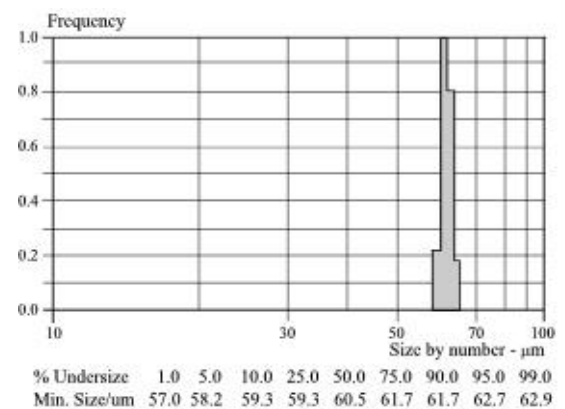


Fig. 9: Microscope analysis of a 63 μm wire woven sieve – minimum dimension of each aperture

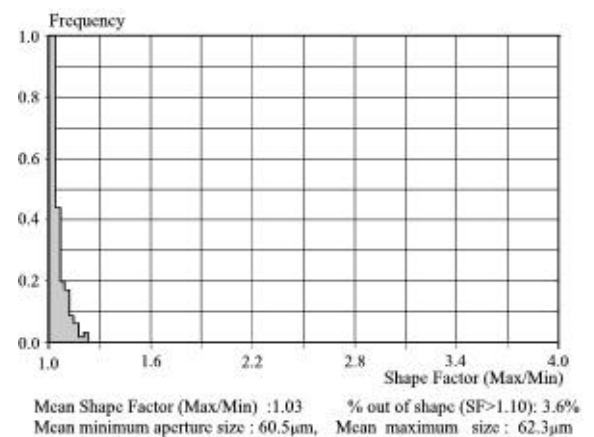


Fig. 10: Aperture shape analysis of a 63 μm wire woven sieve – ratio of mid point dimensions of each aperture

percentage of the total sieve surface. For example, a 200mm diameter 63 μ m test sieve contains nearly 3million apertures. 3000counts, a very time consuming process, would only correspond to a mere 0.1% of the available openings. By contrast, 1g of a 63 μ m sieve calibration standard contains nearly 2.5million beads, so over 80% of the sieve surface is measured producing an unambiguous dimension - the mean aperture size.

Furthermore, because the size distribution of the sieve standard is so narrow, a 5% difference in the percent passing the sieve only results in a mean aperture difference of about 1 μ m. This is reflected in the uncertainty of the measurement - only 0.7 μ m for all 24 analyses performed in this work. This novel approach to sieve calibration using narrow size distribution microspheres therefore fulfils all the requirements of the quality control laboratory - simplicity, speed, accuracy, repeatability and above all, traceability to the International unit of length.

6. References

- (1) G R Rideal Absolute Precision in Particle Size Analysis, American Laboratory, Nov. 1996
- (2) G R Rideal et al, New Reference Standards for Particle Size Instrument Calibration, World Congress on Particle Technology 3 (IChemE) July 1998 Brighton, U.K
- (3) International Standards - ISO 3310-1 (1990), see also BS 410:196 and ASTM E11:87
- (4) R Wilson, K Leschonski, W Alex, T Allen, B Coglin, Certification Report on Reference Materials of Defined Particle Size; Commission of the European Communities; BCR Report: EUR 6825 EN, 1980
- (5) F G Carpenter and V R Deitz, Glass Sphere for the Measurement of Effective Opening of Test Sieves, J. Res. NBS 47, 139 (1951) - now NIST
- (6) T Alan, and A A Khan, Critical Evaluation of Powder Sampling Procedures, The Chemical Engineer, May 1970
- (7) Whitehouse Scientific specially constructed 100-stage spinning riffler, see refs 1,2 and 8 for performance results
- (8) S Dutton, P J Lloyd, Analysis of Riffler Performance, Loughborough Consultants Ltd, July1994 (Confidential Document for BCR
- (9) Gilson Co. Inc. Worthington, Ohio, U.S.A
- (10) Buckbee Mears Co, St Paul, Minnesota, U.S.A
- (11) Shapersizer - Microscope and Image Analyser developed according to BCR guideline methods.
- (12) National Physical Laboratory, Teddington, Middlesex, U.K
- (13) Fritsch AG, Idaar Oberstein, Frankfurt, Germany
- (14) Hozokawa Micron, Runcorn, U.K

Authors Christian names and academic titles

Dr Graham Robin Rideal, BA, M.Sc, Ph.D, C.Chem, MRIC

Mr Jamie Storey, B,Sc

Mr Thomas Robert Morris, BA Hons