Whitehouse Scientific
Setting the Standard since 1983

NIST Traceable

Polydisperse Particle Standard

1-10µm

Part Number: PS194
Nominal Weight: 0.50g x 10 bottles

DISPERSION NOTES:
Samples dispersed for 5 minutes in an ultrasonic bath using a 0.1% deionised aqueous solution of sodium hexametaphosphate.

Notes:
(1) Traceability - Coulter methods - the following were specified - NIST or BCR primary reference standards Microscopy - NIST calibrated stage reference graticule, ref. test no. 821/263573-00 or National Physical Laboratory (Teddington, UK) stage reference graticule - ref. 08A032/930082/82-153.Sieve Analysis - Electroformed sieves calibrated against NIST or NPL graticules. Sedimentation - does not require linear calibration - Stokes' Law used to calculate the hydrodynamic diameter is a fundamental law of physics. Weight traceable through 1g standard - eg NAMAS Laboratory 0134, Certificates T08243 and T11002, recertified weight serial number 181320295 - supplied by Analar.
(2) Minimum of 5 analyses per laboratory for the Sedimentation and Coulter methods. 5000 counts for microscopy.
(3) For a summary of analytical methods see - Rideal G R, Dodds J A & Pons M-N, Leschonski K, Lloyd P J, and Mercus H G, The Development of New Reference Standards for Particle Size Instrument Calibration, World Congress on Particle Technology 3 (IChemE) July 1998, Brighton, UK.
(4) This certificate is only valid if a complete single-shot bottle is used in the analysis.
(5) Whitehouse Scientific Ltd does not accept responsibility for losses, financial of otherwise which may occur as a result of the interpretation or use of the information contained within this certificate.

Certificate of Analysis

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Whatever the particle sizing method...we have the perfect solution! www.WhitehouseScientific.com

NIST TRACEABLE

POLYDISPERSE PARTICLE STANDARD

SODA-LIME GLASS MICROSPHERES

1. Detailed Review

(results by weight/volume, total of 75 individual tests)

Method	Lab	Percentile 10	25	50	75	90
Andreasen Pipette (by weight)	A	2.90	3.53	4.36	5.40	6.59
	I	2.86	3.41	4.13	5.03	6.05
	X	2.69	3.27	4.02	4.95	6.00
	Y	2.66	3.27	3.99	4.80	5.67
	Mean	**2.78**	**3.37**	**4.13**	**5.05**	**6.08**
	SD*	0.10	0.11	0.15	0.22	0.33
Coulter (by weight)	M	2.89	3.37	4.07	5.01	6.21
	J	2.94	3.47	4.24	5.22	6.46
	R	2.89	3.38	4.12	5.13	6.37
	X	2.93	3.40	4.08	5.03	6.09
	W	3.04	3.55	4.28	5.24	6.43
	S	2.97	3.57	4.53	5.21	6.42
	Mean	**2.94**	**3.46**	**4.22**	**5.14**	**6.33**
	SD*	0.05	0.08	0.16	0.09	0.13
Final Mean size	$\bar{x}_{(n=10)}$	**2.88**	**3.42**	**4.18**	**5.10**	**6.23**
***SD (population)**	xs_{n}	0.11	0.10	0.16	0.16	0.26
SD (sample)	xs_{n-1}	0.12	0.11	0.17	0.17	0.28
Uncertainty (95% confidence)		**0.24**	**0.22**	**0.34**	**0.34**	**0.56**

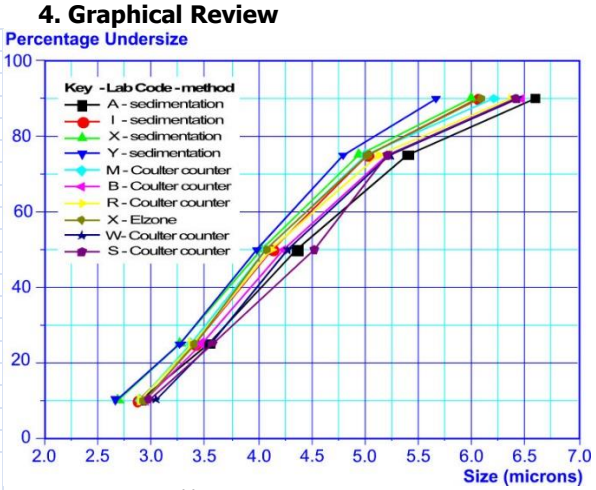
2. Tabular Summary

Cumulative percent undersize	10	25	50	75	90
Final Mean Size – microns	**2.88**	**3.42**	**4.18**	**5.10**	**6.23**
Uncertainty – microns (95% confidence)	0.24	0.22	0.34	0.34	0.56

3. Participating Laboratories

Bradford University, UK
HSE, Sheffield, UK
Whitehouse Scientific, UK
Clausthal TU, Germany
Loughborough University, UK
Delft TU, Holland
Pavia University, Italy

4. Graphical Review



Issued by:

Dr G R Rideal
Founder & Senior Analyst

PRIMARY METHODS OF PARTICLE SIZE ANAYLSIS FOR WHITEHOUSE SCIENTIFIC POLYDISPERSE STANDARDS

ACCURATE SUB-DIVISION – Dr G Rideal, Whitehouse Scientific, UK

All polydisperse particle size standards have been produced using a specially designed 100 stage spinning riffler. The method ensures each bottle of standard is, within experimental error, identical in particle size distribution. All weights are nominal with a standard deviation of approximately 5% (uncertainty 10%).

GRAVITATIONAL SEDIMENTATION – Prof K Leschonski, Germany

To measure the 3-30μm standard, the sedimentation height in the Andreasen Pipette was set to 20cm and extraction times calculated to give 13 particle size points from 29.3μm down to 3.7μm (4 minutes to over 2 hours respectively). The entire contents of the 1g vial was used for the analysis. The device was submerged at 20^{O}C in a water bath controlled to +/- 0.1^{O}C.
Cumulative percent undersize points were plotted for 5 repeat analyses, from which mean sizes at the selected percentiles were calculated. In the case of the 1-10μm standard, a settling height of 10cm was used; 7 extraction times from 14 minutes to approximately 6 hours corresponded to sizes from 10.1μm to 1.5μm respectively.

ELECTROFORMED SIEVE ANALYSIS – Mr H Mercus, the Netherlands

Electroformed nickel sieves having square apertures accurate to +/-2 microns were specified for this method, which was based on ISO 3310-3 (1990). The sieves were calibrated by microscope using the NIST and National Physical Laboratory (NPL) graticules. The height and width of six apertures in 9 measurement fields across the sieve were measured.
Sieve frame dimensions could vary from 70 - 200mm. Any method of mechanical shaking could be used, but the end point was determined by hand shaking when less than 5mg per minute of the initial 10g sample should pass each sieve. The sample loss at the end of the analysis should be less than 0.5% of the charge (ie. 50mg). Five analyses were performed from which the sizes at the prescribed percentiles were calculated.

MICROSCOPY AND IMAGE ANALYSIS – Prof. L Dodds, France

Microscopy and Image Analysis were specified for the 3-30μm, 10-100μm and 150-650μm powders. It was recommended that slides should be prepared from the 1g samples by extracting single drops from a concentrated well-mixed glycerol paste. 35 slides were prepared, 15 fields per slide were counted and 10-20 particles per field analysed. This gave an average count per analysis of about 8,000 particles.
Touching particles were noted but no attempt was made at numerical separation. However, if the total number of touching particles exceeded 20%, the data was discarded and a new slide was prepared. Distortions of camera/microscope lenses were measured using the NPL graticule and limited to 5%. Edge detection was determined by setting the grey scale of the particle to black and the background to white so that, when set to two shades of grey, the computer automatically defined the particle perimeter during scanning. The Miles-Lantuejoul correction was implemented to account for particles cut by the edge of the video frame.

COULTER COUNTER – Dr J Lloyd, UK

The Electrical Sensing Zone (ESZ) method was employed for the 1-10μm, 3-30μm and 10-100μm samples. Like the optical microscope method, the weight of the powder involved could be as low as 5mg so particular attention was paid to the subdivision. A stock suspension was prepared from the 1g bottle of sample using the dispersant sodium hexametaphosphate as electrolyte (1g sample in 400ml of a 5% solution).
In the case of the 10-100μm sample, 40% glycerol / 60% distilled water was used to reduce sedimentation during analysis. By careful weighing, the stock suspension concentration was accurately calculated. A sub-sample (5 or 10ml) was then taken from the rapidly stirred suspension and injected into a known volume of electrolyte in the instrument sample beaker. Thus the sample concentration, and so volume of particles in the measuring siphon of the mercury manometer could be calculated. This predetermined particle volume should not vary by more than 5% from that calculated internally by the instrument. 100,000 particles were analysed and the experiment repeated 5 times for the same beaker of suspension. Data was accumulated for a further 4 dispersions giving a particle count of 2.5 million per sample. The instruments were calibrated using traceable reference standards.