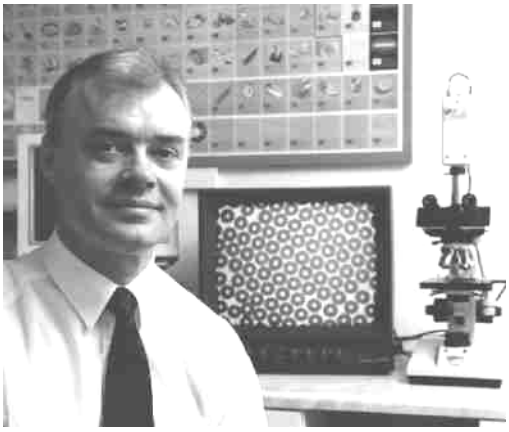
European Success

One UK certification laboratory has been selected on all five primary particle sizing methods

Summary

This report describes the development of a new series of spherical particle size reference standards from the Bureau of Certified Reference (BCR). A duplicate set of standards was produced by Whitehouse Scientific in order to develop prescriptive methods of analysis and to short list about 40 European laboratories who expressed an interest in certifying the new BCR standards. Whitehouse Scientific was the only laboratory to achieve a top 5 placing in all the specified methods.



In a recent round robin exercise sponsored by the Bureau of Certified References (BCR) of the European Commission, Whitehouse Scientific emerged as the leading certification laboratory.

Out of 40 candidates, it was the only laboratory to be selected on all five of the primary particle sizing methods where the dimensions of length and weight are directly traceable to International Standards and do not depend on second-order effects, such as diffraction patterns, turbidity, Brownian motion or computer modelling.

Five European experts were nominated by the commission to prepare prescriptive guide-line methods for each technique:

1. Professor K. Leschonski (Clausthal TU) - for the Andreason pipette sedimentation method;
2. Dr J. Llyod (Loughborough University) - for the electrical sensing zone method;
3. Dr H. Mercus (Delft TU) - for electroformed sieve analysis;
4. Professor J. Dodds (CNRS, Nancy, France) - for optical microscopy and image analysis;
5. Dr G. Rideal (Whitehouse Scientific) - for the sub-micron centrifugal analysis.

The most over-subscribed methods were the electrical sensing zone method and image analysis where about 20 laboratories declared an interest in each.

BCR decided that such a high level of interest would be too much of a drain on the 10kg master batches that had been commissioned (each laboratory had to perform five analysis on every grade) and so Whitehouse Scientifc was asked to prepare and sub-divide a duplicate set of the spherical references for the round robin exercise. The following grades were made and distributed to each candidate laboratory: 0.1- 1μm; 0.3-3μm; 1-10μm; 3-30μm; 10-100μm; and, 150-650μm.

The results were sent directly to the organisers of the programme, AEA Technology, Harwell, where they were coded before being statistically analysed for both accuracy and reproducibility.

Thus the final selection was totally anonymous. Very few laboratories entered all five methods of analysis, but Whitehouse Scientific was the only laboratory that was successful in every category.

A second and equally important aspect of the programme was to check the performance of the 100-stage Whitehouse spinning rifflers that were to be used on the official standards. The advantage of such large rifflers was that the 10kg master batches could be reduced to 1g samples in two stages, minimising handling errors.

The performance of the 100 stage riffler was independently assessed by Loughborough University, and, as a result, Whitehouse Scientific was also contracted to sub-divide 10 out of the 12 grades, making the laboratory the largest single contributor to the BCR programme by a substantial margin.

The need for a new set of spherical reference standards ironically came soon after the BCR introduced its first set in the late 70's. These were produced from crushed quartz and were greater than 1μm in size. Grades below 100μm were measured by the Andreason pipette sedimentation method while the largest grade (150-650μm) was certified using electroformed sieves. Loughborough and Clausthal were also involved in this original programme.

Concurrent with the production of the quartz reference standards was the emergence of laser diffraction methods of particle-size analysis. The quartz standards were both irregular in shape and semi-transparent, the combined effect allegedly accounting for some of the large differences observed in some of the earlier laser particle size-analysers.

It was therefore decided that any new standards should be spherical in shape, non-porous and available both in transparent and opaque variants.

The International Fine Particle Research Institute (IFPRI) headed by L. Ford, was instrumental in the original specifications and commissioned Professor Iinoya of Kyoto University, Japan, to make the +1μm grades. Dr D. Booker of AEA Technology, Harwell, was to make the sub-micron grades.

So far, over 50,000 samples of the Japanese grades have been sub-divided by Whitehouse Scientific and are about to be sent to the successful certification laboratories by AEA Technology. The sub-micron grades have not yet been produced. It is hoped that the official standards will be certified by the end of 1997.

In addition to a complete set of supporting standards (known as 'mirror' standards), there have been several important spin-offs from the BCR programme including an image analysis programme designed by Whitehouse Scientific based on Professor Dodd's guidelines - Shapesizer.

Finally, there has been such a high international response to the proposed new reference standards from particle metrologists that the original Whitehouse range has been extended to include sieve calibration standards and monodisperse microspheres.