

Does your scale bar measure up?

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Electron and scanning-probe microscopies have long been at the heart of nanoscience, with many innovations only becoming possible with advances in the corresponding microscopy technology. As nanoscience matures from the lab to commercially realised technologies, microscopy continues to play a critical role. The nanotechnology community needs nanoscale measurements that are accurate, internationally recognised and relevant for the intended applications: researchers need to calibrate or verify their measuring instruments, industry must compete with their products in national and international markets whilst complying with regulations, governments need to establish or interpret regulatory requirements, and consumers demand to know what their products contain.

Physical standards, traceable to internationally agreed references along with documentary standards that specify validated methodologies, provide the confidence in measurement results that the nanotechnology community needs. Here, we give an update on activities at the National Measurement Institute (NMI) which are directly related to improving the confidence users can have in nanoscale dimensional measurements.

Firstly, to ensure the comparability and international recognition of nanoscale dimensional measurements, NMI has developed a Metrological Scanning Probe Microscope, which links the SI metre to the nanometre. This is a specialised precision instrument based on heterodyne laser interferometry which monitors the displacement of a scanning stage in three dimensions in feedback to a quartz crystal tuning fork force sensor.

We also present the contribution made by NMI to the development of certified reference materials for calibration of electron and scanning probe microscopes, released by the European Commission's Joint Research Centre. We also discuss NMI's participation in work carried out in Technical Committee 229 "Nanotechnologies" of the International Organization for Standardization (ISO) that aims to develop validated protocols for the measurement of primary particle size distributions by transmission electron microscopy (TEM) and will lead to a corresponding ISO standard. NMI's involvement here has focused on silica (analysed using TEM and atomic force microscopy as well as several ensemble techniques), gold nanorods and titania samples (TEM analysis only).

This work highlights the challenge of measuring the dimensional properties of many thousands of individual particles to achieve statistical relevance, the influence of image analysis parameters and their effects on the measurement uncertainty, and the challenges of measuring non-spherical particles. We also discuss the issue of method divergence, i.e., differences in measurement results between different measurement techniques, and provide insight into measurement uncertainty contributions arising from a number of different influence factors.