

Accurate powder diffraction standards: determination of the lattice parameter of LaB₆ (standard reference material 660)

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We use X-ray powder diffraction and synchrotron radiation to determine the lattice parameter of the NIST standard reference material (SRM 660) LaB₆ to be 4.157580 Å with an accuracy of 12 parts per million (ppm), calibrated relative to the lattice parameter of the Si powder standard ($a_0 = 5.430940(11)$ Å, Si 640b). A discrepancy is observed between the currently accepted lattice spacing of LaB₆ and the measured value, of 0.00048(5) Å, or nine standard deviations from the NIST reference. Twelve different measurements of the lattice parameter are made at beam energies between 10 keV and 20 keV. The observed discrepancy in the lattice parameter is consistent for the different energies used.

The absolute values of the mean difference between the measured and calculated 2 theta centroids, are highly consistent, between 0.0002° and 0.0004° for energies from 5 keV to 14 keV, and between 0.0005° and 0.0008° for energies from 15 keV to 20 keV. In order to determine the peak positions with high precision, account must be taken of observed peak asymmetry. Significant asymmetry is due to peak broadening and must be taken into account in order to determine accurate peak locations and lattice spacings.

Our approach shows significant advantages over conventional analysis. Our analysis of peak broadening is compared with models used in Rietveld analysis.