

NIST Standard Reference Materials for X-ray Metrology

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The powder diffraction method has an exceptionally broad range of applications owing to its relative ease of use and its applicability to any crystalline material. Furthermore, powder diffraction can access a considerable breadth of sample characteristics: qualitative and quantitative phase analysis as well as crystal- and micro-structure determinations. Each application presents a specific challenge with regards to the development of a standard, both in the context of the character of the standard itself and the method by which it is certified. The goal is to provide a standard that will link the measurands to the *Système international*, SI, in a traceable manner.

The certification of a NIST SRM involves: 1) microstructure engineering and/or evaluation to obtain an SRM artifact properly suited to address the measurement issue at hand, 2) the design and commissioning of the equipment that embodies the unique features required for SI traceable measurements, and 3) the design of experiments, measurement methods and data analysis strategies that allow for a valid assessment of systematic uncertainties of the certified measurement quantities. A fourth component of the program entails the examination of the methods whereby an SRM is used in the field for calibration of instruments and measurements.

Obtaining SRM feedstock of an optimal microstructure can often be quite difficult. The desired route is to utilize industrially mature processes, tailored to fit our needs, to manufacture the feedstock through dedicated processing runs vis commercial vendors. SRMs 660c, 1976b, 1879b, 1878b and 640e are examples of such SRMs. SRMs 674b and 656 are commercially available powders selected for their suitability to serve as SRMs. Certain SRMs are made entirely in house, or are processed to some degree in house, in a manner not available commercially. The two zinc oxide powders of SRM 1979 were manufactured entirely in house. Zinc oxalate was decomposed in a NIST-built vacuum furnace to yield two powders; these were further annealed in air to yield crystallites in the 15 nm and 60 nm size range.

X-ray diffraction measurements on SRMs certified with respect to lattice parameter are performed on the NIST-built divergent beam diffractometer, DBD. While this diffractometer includes several unique features that enable it to provide SI traceable measurements, we have also commissioned a parallel beam diffractometer, PBD, that includes additional features which will yield measurements with reduced error bounds. We report on preliminary results from the PBD, comparing these results from those of the DBD. SRM 1979 was certified using high-resolution synchrotron data from 11BM while laboratory data are provided for informational purposes.

SRMs certified for quantitative analysis are typically certified using neutron, constant wavelength and time-of-flight powder diffraction data. Such data are not prone to the systematic errors that affect the results from conventional laboratory sources. The homogeneity testing and certification of lattice parameters will nonetheless be performed using laboratory data.