

Renewed and Upgraded NIST SRMS for Powder Diffraction

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SRM 1879a, Respirable Cristobalite (Quantitative X-Ray Powder Diffraction Standard), has been renewed as SRM 1879b. SRM 1879b was certified for quantitative analyses of cristobalite in accordance to guidelines from the National Institute for Occupational Safety and Health; i.e., Analytical Method 7500. The cristobalite feedstock was prepared with a high-temperature anneal of vitreous silica. High purity, 99.995%, silica was annealed at 1600 °C for two hours in an induction furnace. The material was then jet milled to a median particle size of 3.5 μm. It has been certified with respect to phase purity, or amorphous content, and lattice parameters. The feedstock was also verified to be homogeneous with respect to diffraction properties.

Phase purity was certified with the Rietveld analyses of time-of-flight data from POWGEN (ORNL) and constant wavelength data from BT1 (NCNR, NIST). SRM 676a, certified with respect to phase purity, was used as the internal standard. The diffraction experiment measures only the mass of crystalline material, while the weighing operation used in sample preparation is non-discriminatory. The discrepancy between the mass fractions admixed during sample preparation and the diffraction experiment is indicative of the amorphous content of the “unknown” phases within the mixture. Correlation between results from fundamentally dissimilar diffraction geometries lends credibility to the results. X-ray data were collected on a NIST-built diffractometer that features several attributes specific to flexibility and accuracy. Data were analyzed with the fundamental parameters approach using both the Pawley and Rietveld methods. These results were used to verify homogeneity and certify the lattice parameters.

It was discerned that lattice parameter refinements from mixtures can lead to biased results. The lattice parameters of SRM 1878b, Respirable Quartz, were recertified using phase pure samples.

SRM 674b, X-Ray Powder Diffraction Intensity Set (Quantitative Powder Diffraction Standard), consisting of four powders, ZnO, TiO₂, Cr₂O₃, and CeO₂, was originally certified in September of 2005. The lattice parameters were reported as non-certified “informational values”. However we now understand the goniometer assembly used for the initial measurements was flexing leading to errors in the 50 fm to 10 fm range. We report on the certification of lattice parameters of SRM 674b. New values are retroactive to units in the field.

SRM 656, Silicon Nitride Powders (Quantitative Analysis Powder Diffraction Standard) was originally certified in 1995. It consists of two powders of the two polymorphs of silicon nitride. One powder is high in α phase content, while the other contains a larger amount of the β polymorph. We revisit the certification of this SRM, certifying the lattice parameters and updating the relative intensity and RIR values to those determined with modern methods.