National Institute of Standards & Technology

Certificate

Standard Reference Material® 640f

Line Position and Line Shape Standard for Powder Diffraction
(Silicon Powder)

This Standard Reference Material (SRM) is intended for use as a standard for calibration of diffraction line positions and line shapes, determined through powder diffractometry. A unit of SRM 640f consists of approximately 7.5 g of silicon powder bottled under argon.

Material Description: The SRM was prepared from ultra-high purity, intrinsic silicon boules that were crushed and jet milled to a median particle size of 4.1 μm. The resulting powder was then annealed under gettered argon at 1000 °C for two hours [1] and bottled under argon. Analysis of X-ray powder diffraction data indicated that the SRM material is homogeneous with respect to diffraction properties.

Certified Value: The certified lattice parameter for a temperature of 22.5 °C is

0.543 114 4 nm ± 0.000 008 nm

The interval defined by this value and its expanded uncertainty ($k = 2$) is dominated by a Type B uncertainty estimated from a technical understanding of the measurement data and its distribution. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account. The certified values and uncertainties were calculated according to the method described in the ISO/JCGM Guide [2]. The measurand is the lattice parameter. Metrological traceability is to the SI unit of length, expressed as nanometers.

Information Values: The analyses of the certification data included refinement of the full-width half-maximum (FWHM) of a Lorentzian profile to account for sample-induced broadening. The angular dependence of the FWHM term varying as $1/\cos\theta$ is interpreted as size-induced broadening. The value obtained was consistent with a mean volume-weighted domain size of approximately 0.4 μm. The term varying as $\tan\theta$, interpreted as microstrain, refined to zero. The information values for computed peak positions are given in Table 1. The typical particle size distribution as determined by laser scattering is given in Figure 1. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 640f** is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage"). Periodic recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall coordination and technical direction of the certification were performed by J.P. Cline of the NIST Materials Measurement Science Division.

The preparation, measurements and data analyses were performed by J.P. Cline, M.H. Mendenhall, D. Black and E.G. Kessler of the NIST Materials Measurement Science Division and A. Henins of the NIST Quantum Measurement Division.

R. David Holbrook, Acting Chief
Materials Measurement Science Division

Gaithersburg, MD 20899
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Steven J. Choquette, Director
Office of Reference Materials

Collection of the laser scattering particle size data for the informational values was performed by M. Peltz of the NIST Materials and Structural Systems Division.

Statistical analysis was by provided J.J. Filliben of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR STORAGE

SRM 640f was bottled under argon to protect against humidity. When not in use, store the unused portion of this powder tightly capped in the original bottle or in a manner with similar or greater protection against humidity.

SOURCE, PREPARATION, AND ANALYSIS[1]

Source of Material: The silicon was obtained from Siltronic AG (Munich, Germany). The comminution was performed by Hosokawa Micron Powder Systems (Summit, NJ).

Certification Method: Certification was performed using data from a NIST built diffractometer [3], with analyses via the fundamental parameters approach (FPA) [4] using the Pawley method [5]. These analyses were used to verify homogeneity and certify the lattice parameters. The linkage of the certified lattice parameter values to the fundamental unit of length, as defined by the International System of Units (SI) [6], was established with use of the emission spectrum of Cu Kα radiation as the basis for constructing the diffraction profiles. With the use of the FPA, diffraction profiles are modeled as a convolution of functions that describe the wavelength spectrum, the contributions from the diffraction optics, and the sample contributions resulting from microstructural features. Analysis of data from a divergent-beam instrument requires knowledge of both the diffraction angle and the effective source-sample-detector distance. Two additional models are therefore included in the FPA analyses to account for the effect of the sample height and attenuation. Certification data were analyzed in the context of both Type A uncertainties, assigned by statistical analysis, and Type B uncertainties, based on knowledge of the nature of errors in the measurements, to result in the establishment of robust uncertainties for the certified values.

The uniformity of the single-crystal silicon material was verified prior to comminution. These measurements were performed on the NIST lattice comparison apparatus [7] using 11 crystal samples taken from the supplied material. A total of 32 lattice comparison measurements covering the longitudinal and radial boule directions were made. The relative lattice variation indicated by these measurements was $\pm 4.8 \times 10^{-8}$ (95 % confidence level) indicating the material was sufficiently uniform for use as a powder line position SRM to be certified with respect to lattice parameter [8].

Certification Procedure: Ten units of SRM 640f were selected in a stratified random manner from the population of units during the bottling operation. Certification data were recorded from 2 samples prepared from each of 10 bottles, for a total of 20 samples. The optical layout of the diffractometer used for the certification measurements was that of a conventional divergent-beam diffractometer of Bragg-Brentano geometry, equipped with a Johansson incident beam monochromator (IBM), sample spinner, and a position sensitive detector (PSD). The 1.5 kW copper tube of fine focus geometry was operated at a power of 1.2 kW. The variable divergence incident slit was set to 0.9°. A 1.5° Soller slit was located in front of the PSD window to limit axial divergence, no Soller slits were used in the incident beam. Data were collected from a 2θ range of 25° to 140°; the scan time was approximately 2.5 hours. The PSD was scanned in "picture taking" mode wherein data from the full length of the PSD window were recorded. With a window length of 14.4 mm, divided in to 192 strips of 75 μm, and a goniometer radius of 217 mm, this corresponds to window dimension 3.8 °2θ, and an angular resolution of 0.02 °2θ. A combination of coarse steps, in the context of 3.8°, and fine ones, in the context of 0.02 °2θ, was used; this allowed for timely data collected at high resolution. Post data collection processing allows for scaling the PSD window length with Tanθ. The result is a pattern which effectively has variable dwell time, resulting in improved statistics for the high-angle reflections, at no cost to the resolution [9]. The machine was equipped with an automated anti-scatter slit located above the sample to prevent air scatter from the incident from entering the PSD and contributing to the low angle background level. Its height above the specimen varied as $\alpha R / (2 \cos\theta)$ where α is the full equatorial divergence angle of the incident beam. The machine was located within a temperature-controlled laboratory space where the nominal short-range control of temperature was ± 0.1 °K. The temperature was monitored using two 10 kΩ thermistors with a Hart/Fluke BlackStack system that was calibrated at the NIST temperature calibration facility [10] to ±0.002 °C. The source was equilibrated at operating

[1]Certain commercial equipment, instruments, or materials are identified in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

conditions for at least an hour prior to recording any certification data. The performance of the machine was qualified with the use of SRM 660c *Line Position and Line Shape Standard for Powder Diffraction* [11] and SRM 676a *Alumina Powder for Quantitative Analysis by X-Ray Diffraction* [12] using procedures discussed by Cline *et al*. [3].

Data Analysis: The certification data were analyzed using the FPA method with Pawley refinements as implemented in TOPAS [13]. Mendenhall *et al.* [14] verified that TOPAS operated in accordance with published models for the FPA. The analysis used energies of the Cu Kα emission spectrum as characterized by Mendenhall *et al*. [15]. The optics of the Johansson IBM were modeled using dynamical scattering from the monochromator in conjunction with the powder sample as per the optics of a 2-crystal monochromator. The resulting "band pass" model provides a "window" function which modulates the intensity of the native copper emission line from our x-ray tube, effectively cutting off the Lorentzian tails of the native lines, providing good agreement with the shape of the tails of the diffraction peaks. It also adds a dispersion term to the FPA emission model which adds to the width of the modeled lines, further improving the fit to the observation [16]. Parameters associated with the bandpass model, as well as others of the IPF, the incident slit angle and the Soller slit angles of the "full" axial divergence model [17] were refined using scans from SRM 660c. They were then fixed at the SRM 660c values for the analyses of SRM 604f. Other refined parameters included the scale factors, Chebyshev polynomial terms for modeling of the background, the lattice parameters, specimen displacement and attenuation terms and a term for Lorentzian size broadening. The refined lattice parameters were adjusted using the CTE values found in Schödel & Bönsch, [18] to values at 22.5 °C.

A statistical analysis of the data indicated that the mean of the measurements was 0.543 114 4 nm with a $k = 2$ Type A expanded uncertainty of 0.000 000 54 nm. However, a Type B uncertainty due to systematic error must be incorporated into the uncertainty bounds of the certified lattice parameter. Consideration of trends in the data used in the certification leads to an assignment of a Type B uncertainty and value as stated on page 1.

Table 1. Information Values for Peak Positions Computed for SRM 640f Using Cu Kα Radiation, λ = 0.15405929 nm

h	*k*	*l*	2θ (degrees)
1	1	1	28.441
2	2	0	47.301
3	1	1	56.120
4	0	0	69.127
3	3	1	76.373
4	2	2	88.026
5	1	1	94.947
4	4	0	106.702
5	3	1	114.085
6	2	0	127.535
5	3	3	136.882

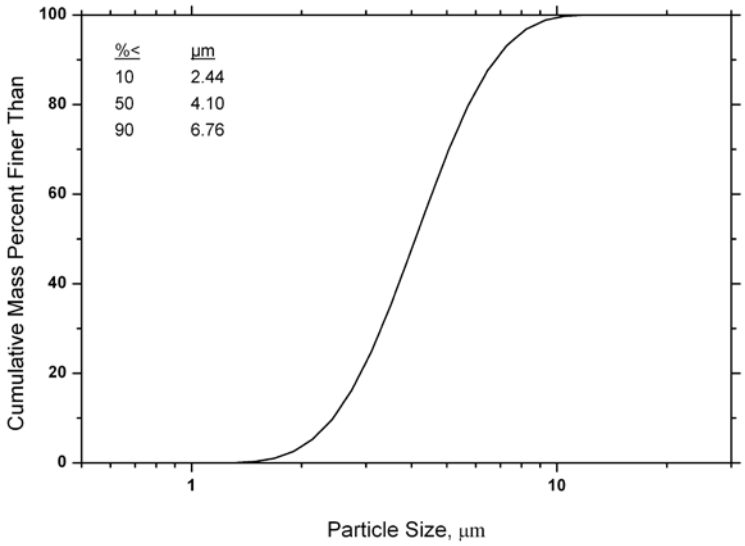

Figure 1. Typical Particle Size Distribution as Determined by Laser Scattering

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Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.