

National Bureau of Standards Certificate Standard Reference Material 640 Silicon Powder Lattice Parameter (X-Ray Diffraction Standard)

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This silicon powder Standard Reference Material was prepared for use as an internal standard in powder diffractometry.

Electronic grade float-zone prepared silicon boules from which this SRM was prepared were 99.999+% pure before grinding. The grinding reduced the purity to 99.9% by the addition of surface contaminants. The powder obtained as a result of this grinding (SRM 640) passed a 200 mesh sieve (75 μm) and consists primarily of 10 μm particles although some particles are as large as 100 μm . In certain cases further sieving of this SRM may be appropriate.

A total of twenty-one samples, mixed with a tungsten internal standard [1], were measured using a high angle goniometer. The observed diffraction angles were corrected through use of the internal standard. Each corrected set was refined by a least-squares routine that minimized $\Sigma (2\theta_{\text{obs}} - 2\theta_{\text{calc}})^2$ to obtain estimates of a_i and their standard errors s_i . The weighted average, \bar{a} , and the standard error, s , were calculated from the twenty-one values.

The weighted average of the lattice parameter, \bar{a} , uncorrected for refraction is 5.43088Å on the absolute wavelength scale of Deslattes and Henins. [2] The standard error of the average is estimated to be 3.5×10^{-5} Å. It includes contributions from three sources (listed in decreasing importance): (1) uncertainty of the lattice parameter of the tungsten standard; (2) random errors of measurement; and (3) the uncertainty in λ . Comparison of \bar{a} with single crystal values from the same source reveals a difference of 3 parts in 10^5 . [3]

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Diffraction Angles for SRM 640

$$a = 5.43088 \text{ \AA}$$

$$\lambda = 1.5405981 \text{ \AA}, T = 25.0 \text{ }^\circ\text{C}$$

<u>hkℓ</u>	<u>2θ</u>	<u>hkℓ</u>	<u>2θ</u>
111	28.443°	511/333	94.954°
220	47.303	440	106.710
311	56.123	531	114.094
400	69.131	620	127.547
331	76.377	533	136.897
422	88.032	444	158.638

When using SRM 640 with a goniometer and interpolating angles to 0.0025° , the measurement should be reproducible to 2 parts in 10^5 . Results based on this SRM will be consistent with the NBS results in the Powder Diffraction File (when converted to the same wavelength), which were based on the NBS internal standards of 1966. [1]

- [1] Swanson, H. E., McMurdie, H. F., Morris, M. C., and Evans, E. H. (1966), Standard X-Ray Diffraction Powder Patterns, National Bureau of Standards Monograph 25 - Section 6, NBS, Washington, D. C. 20234.
- [2] Deslattes, R. D. and Henins, A. (1973), Phys. Rev. Letters, 31, 972-975.
- [3] Hubbard, C. R., Mauer, F. A. and Swanson, H. E., (1974). J. Appl. Cryst. - submitted for publication.

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