S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 640a

Silicon Powder 2θ/d-Spacing Standard for X-Ray Diffraction

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This Standard Reference Material (SRM) was prepared for use as an external or internal 2θ/d-spacing calibration standard for powder diffractometry.

Electronic grade float-zone prepared silicon boules, 99.999 +% pure, were ground to pass a 75 μm (200 mesh) sieve. This powder, which was identical to SRM 640, was then jet-milled to reduce the mean particle size to about 2 μm.

A total of twelve samples, mixed with tungsten and silver internal standards [1], were measured using a high angle goniometer controlled by a minicomputer. The CuKα_1 peak position was determined by profile fitting procedures and then corrected for sample, instrumental, and physical aberrations (except refraction) through use of the internal standard lines [2]. Silicon peak positions were then corrected for effects of thermal expansion. Each corrected set was refined by a least-square routine that minimized $\Sigma(\theta_{obs}-\theta_{calc})^2$ to obtain estimates of a_i and their estimated standard errors, s_i. The weighted average of the twelve lattice parameters, uncorrected for refraction, at 25 °C is

$$\langle a \rangle = 5.430825 \pm 0.000036 \text{ Å}$$

where $\lambda(\text{CuK}\alpha_1) = 1.5405981$ Å [3]. The estimated total uncertainty given above includes contributions from three sources (listed in decreasing importance): (1) uncertainty of the lattice parameters of the tungsten and silver standards; (2) random errors of the measurements; and (3) the uncertainty in $\lambda(\text{CuK}\alpha_1)$.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.K. Kirby.

Washington, D.C. 20234
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George A. Uriano, Chief
Office of Standard Reference Materials

(over)

The 2θ values given in Table 1 were calculated from the certified value of the lattice parameter. The relative intensities [4], I (rel), also given in Table 1 can be used as an aid in identifying the silicon lines when SRM 640a is mixed with test materials. The uncertainty of each value of I (rel) may be as large as $\pm$ 3. Suggested methods for use of this SRM are given in references [2], [5], and [6].

Table 1

Calculated Diffraction Angles and Relative Intensities

(T = 25.0 °C)

These Values Are Not Certified

hkℓ	I (rel)	2θpeak	hkℓ	I (rel)	2θpeak
111	100	28.443°	511/333	6	94.955°
220	55	47.304	440	3	106.712
311	30	56.124	531	7	114.096
400	6	69.132	620	8	127.550
331	11	76.378	533	3	136.900
422	12	88.033	444	*	158.644

*Not measured

[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 4, NBS, Washington, D.C. 20234.

[2] Hubbard, C.R. (1982), submitted for publication in J. Appl. Cryst.

[3] Deslattes, R.D. and Henins, A. (1973), Phys. Rev. Letters, 31, 972-975.

[4] Morris, M.C.; McMurdie, H.F.; Evans, E.H.; Paretzkin, B.; deGroot, J.H.; Hubbard, C.R.; and Carmel, S.J. (1976) Standard X-Ray Diffraction Powder Patterns, National Bureau of Standards Monograph 25, Section 13, NBS, Washington, D.C. 20234.

[5] Snyder, R.L.; Hubbard, C.R.; and Panagiotopoulos, N.C., Advances in X-Ray Analysis, Vol. 25, (in press).

[6] Hubbard, C.R. (1980) Accuracy in Powder Diffraction, NBS Special Publication 567, p 489-502.