U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 640b

Silicon Powder 2θ/d-Spacing Standard

for X-Ray Diffraction

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

SRM 640b is a high-purity silicon powder prepared by grinding electronic grade silicon rods, followed by jet milling to reduce the particle size. The median particle size (d_{50}), based on a mass-weighted distribution as determined by x-ray sedigraph, is about 5 μm; 95 percent of the particles on a mass basis have an average diameter less than 10 μm.

A total of 25 samples was mixed with tungsten and silver internal standards [1] and measured using two automated high angle goniometers. The CuKα_1 peak position was determined by second derivative and profile fitting procedures and was then corrected for effects of thermal expansion. The peak positions were corrected for sample, instrumental, and physical aberrations (except refraction) through use of the internal standard lines [2]. The lattice parameter at 298.1 K of each of the 25 samples was obtained through a least squares refinement of the corrected 2θ values, using NBS*LSQ85, a version of the lattice parameter refinement program of Appleman and Evans [3]. The weighted average of the 25 lattice parameters, uncorrected for refraction, at 298.1 K is

$$\bar{a} = 5.430940 \pm 0.000035 \text{ Å}$$

where $\lambda(\text{CuK}\alpha_1) = 1.5405981$ Å [4]. 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 coordination leading to certification was provided by C.R.Hubbard, with material and sample preparation and technical measurements provided by C. Robbins and W. Wong-Ng, all of the Inorganic Materials Division, Institute for Materials Science and Engineering.

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 L. Kieffer and R. McKenzie.

January 14, 1987
Gaithersburg, MD 20899

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(Over)

The 2θ values given in Table 1 were calculated from the certified value of the lattice parameter and are identified by the appropriate indices (hkl). The relative intensities, I^{rel}, also given in Table 1, can be used as an aid in identifying the silicon lines when SRM 640b is mixed with test materials. Average relative intensities for both randomly oriented samples using a side-drifted sample holder [5] and for tightly packed samples using a front-loaded sample holder are reported [6]. Suggested methods for use of this SRM are given in references [2], [7], [8], and [9].

Table 1

Calculated Diffraction Angles and Relative Intensities

(T = 298.1 K)

These Values Are Not Certified

hkl	I^{rel} Sided Drifted[5]	I^{rel} Front Loaded ≠	2θ peak	hkl	I^{rel} Sided Drifted	I^{rel} Front Loaded	2θ peak
111	100	100	28.442°	511/333	6	8	94.953°
220	55	64	47.303	440	3	5	106.709
311	30	34	56.122	531	7	9	114.092
400	6	8	69.130	620	8	7	127.545
331	11	12	76.376	533	3	3	136.893
422	12	16	88.030	444	*	3	158.632

*Not measured
≠This work

[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, p. 3, NBS, Washington, DC 20234.

[2] Hubbard, C.R., J. Appl., Cryst. (1983), **16**, 285-288.

[3] Appleman, D.E. and Evans, H.T., Jr., (1973). Report #PB216188, U.S. Dept. of Commerce, National Technical Information Service, 5285 Port Royal Rd., Springfield, VA 22151.

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

[5] 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, p. 35, NBS, Washington, DC 20234.

[6] The I^{rel} values correspond to a constant volume of sample contributing to diffraction, independent of 2θ. (For a focusing diffractometer this is achieved by using a fixed angle divergent slit or by correcting for changing volume when theta-compensating divergent slits are used.)

[7] Snyder, R.L.; Hubbard, C.R.; and Panagiotopoulos, N.C., Advances in X-Ray Analysis, (1982), **25**, 245-260, Plenum Press.

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

[9] Hubbard, C.R.; Wong-Ng, W.; and Jenkins, R. (1987), submitted to Powder Diffraction.