Table 5.2.10.4. *Silicon standard high reflection angles (°) (NIST SRM 640c, $a_0 = 5.431195$ Å, $T = 295.6$ K, $\lambda = 0.709317$ Å)*

h	k	l	d (Å)	2θ	h	k	l	d (Å)	2θ
10	6	0	0.46572	99.198	9	9	5	0.39717	126.497
8	6	6	0.46572	99.198	8	8	8	0.39196	129.600
11	3	3	0.46067	100.686	13	5	1	0.38894	131.530
9	7	3	0.46067	100.686	11	7	5	0.38894	131.530
12	0	0	0.45260	103.183	10	10	0	0.38404	134.882
8	8	4	0.45260	103.183	10	8	6	0.38404	134.882
11	5	1	0.44796	104.694	14	2	0	0.38404	134.882
7	7	7	0.44796	104.694	13	5	3	0.38120	136.990
12	2	2	0.44053	107.235	11	9	1	0.38120	136.990
10	6	4	0.44053	107.235	12	8	0	0.37659	140.703
11	5	3	0.43624	108.777	11	9	3	0.37390	143.079
9	7	5	0.43624	108.777	9	9	7	0.37390	143.079
12	4	0	0.42937	111.378	12	6	6	0.36955	147.363
9	9	1	0.42540	112.961	10	10	4	0.36955	147.363
10	8	2	0.41903	115.642	14	4	2	0.36955	147.363
9	9	3	0.41533	117.279	13	7	1	0.36701	150.191
11	7	1	0.41533	117.279	11	7	7	0.36701	150.191
11	5	5	0.41533	117.279	13	5	5	0.36701	150.191
13	1	1	0.41533	117.279	12	8	4	0.36289	155.551
12	4	4	0.40939	120.064	11	9	5	0.36048	159.376
11	7	3	0.40595	121.773	15	1	1	0.36048	159.376
13	3	1	0.40595	121.773	13	7	3	0.36048	159.376
9	7	7	0.40595	121.773	14	6	0	0.35658	168.113
12	6	2	0.40039	124.694					
13	3	3	0.39717	126.497					

5.2.12. Instrumental line-profile-shape standards

The need for standard reference materials to determine instrumental line profiles arose from the increased use in recent years of whole-pattern methods (Section 5.2.6) in several applications of powder diffraction. Instrumental line-profile standards are required to determine resolution, as a check that alignment has been optimized, or to compare the performance of different diffractometers, and to obtain sample contributions from observed data in line-profile analysis. Different standards may therefore be required if samples of interest do not have a high absorption coefficient for the radiation used.

In addition to the usual requirements for SRMs, suitable substances for instrument characterization clearly should not exhibit any measurable sample broadening, even when used with high-resolution diffractometers. Various materials were considered by the Technical Committee of the JCPDS–ICDD, in association with NIST, and lanthanum hexaboride [LaB_6: $a_0 = 4.15695(6)$ Å at $T = 299$ K] was selected for use as an instrumental standard (Fawcett *et al.*, 1988). This was subsequently marketed by NIST as SRM 660 and it also serves as a line position standard. Other materials used as instrumental standards include BaF_2 (Louër & Langford, 1988) and KCl (Scardi, Lutterotti & Maistrelli, 1994). Both are low-cost materials, are available in large quantities, and can readily be annealed to minimize sample broadening. Although KCl introduces a measurable contribution to line breadth owing to sample transparency, it can be used to advantage for correcting data from materials having a similar absorption coefficient, such as many ceramics. van Berkum, Sprong, de Keijser, Delhez & Sonneveld (1995) selected a 5–10 μm size fraction from silicon SRM 640b, deposited about 1.5 Mg m^{-2} uniformly on a (510)-oriented single-crystal silicon wafer and annealed the whole assemblage to produce an instrument line-profile standard. The resulting line-profile widths were found to be slightly less than for LaB_6 at angles below about 100°(2θ) with Cu $K\alpha$ radiation.

5.2.13. Factors determining accuracy

Many factors influencing accuracy in lattice-parameter determination have been mentioned in passing or discussed at length in this and previous chapters. This section attempts to summarize them and put them into perspective. Accuracy in the range of 1 to 0.1% can now be achieved routinely with average care. Increasing the accuracy to 0.01% requires considerable care in specimen preparation, data collection, instrument alignment, and calibration. The range 0.001 to 0.0001% is rarely reached and each determination is virtually a research project. The more important factors are:

(1) Differentiation of the Bragg equation, as in (5.2.1.4), shows the advantage of using the highest-angle reflections; because of the $\cot\theta$ term, the error in Δd is smaller for a given angular accuracy $\Delta\theta$. The gain is not as great as one might expect at first, as the experimental accuracy of the back reflections is lowered because of (i) their lower intensity, (ii) their lower peak-to-background ratio, (iii) their broadening by wavelength dispersion and crystallite imperfection, and (iv) problems of overlapping.

(2) The lower-angle reflections show the converse effects of (i) higher intensity, (ii) higher peak-to-background ratio, (iii) less broadening, and (iv) fewer problems of overlapping. In any particular case, a balance of advantage must be sought.

references