International Tables for Crystallography (2006). Vol. C, Section 5.2.13, pp. 501–504.

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.

Copyright © 2006 International Union of Crystallography

Table 5.2.10.5. *Tungsten reflection angles (°) ($a_0 = 3.16523$ Å, $T = 298$ K)*

h k l	*d* (Å)	*I*	Mo $K\alpha_1$ 0.709317 Å	1.000000 Å	1.250000 Å	1.500000 Å	1.750000 Å	Cr $K\alpha_1$ 2.289746 Å
1 1 0	2.23816	100.0	18.235	25.817	32.431	39.157	46.027	61.531
2 0 0	1.58262	18.1	25.899	36.834	46.521	56.575	67.130	92.672
2 1 1	1.29220	37.0	31.860	45.528	57.851	70.958	85.241	124.747
2 2 0	1.11908	11.1	36.953	53.076	67.903	84.164	102.868	
3 1 0	1.00093	14.4	41.505	59.938	77.279	97.059	121.896	
2 2 2	0.91372	3.3	45.679	66.352	86.316	110.333	146.518	
3 2 1	0.84594	14.2	49.574	72.464	95.262	124.894		
4 0 0	0.79131	1.3	53.255	78.376	104.339	142.810		
3 3 0	0.74605	2.0	56.768	84.164	113.805			
4 1 1	0.74605	4.0	56.768	84.164	113.805			
4 2 0	0.70777	3.1	60.145	89.893	124.027			
3 3 2	0.67483	2.5	63.411	95.621	135.687			
4 2 2	0.64610	2.0	66.586	101.406	150.632			
5 1 0	0.62075	1.6	69.687	107.312				
4 3 1	0.62075	3.2	69.687	107.312				
5 2 1	0.57789	2.2	75.717	119.815				
4 4 0	0.55954	0.5	78.668	126.656				
5 3 0	0.54283	0.8	81.589	134.172				
4 3 3	0.54283	0.8	81.589	134.172				
6 0 0	0.52754	0.2	84.488	142.810				
4 4 2	0.52754	0.7	84.488	142.810				
6 1 1	0.51347	0.6	87.373	153.695				
5 3 2	0.51347	1.2	87.373	153.695				
6 2 0	0.50047	0.5	90.251	175.042				
5 4 1	0.48841	1.0	93.129					
6 2 2	0.47718	0.4	96.016					
6 3 1	0.46669	0.8	98.919					
4 4 4	0.45686	0.1	101.845					
5 5 0	0.44763	0.2	104.802					
7 1 0	0.44763	0.3	104.802					
5 4 3	0.44763	0.7	104.802					
6 4 0	0.43894	0.3	107.800					
5 5 2	0.43073	0.3	110.851					
6 3 3	0.43073	0.3	110.851					
7 2 1	0.43073	0.6	110.851					
6 4 2	0.42297	0.6	113.963					
7 3 0	0.41562	0.3	117.150					
7 3 2	0.40198	0.5	123.837					
6 5 1	0.40198	0.5	123.837					
8 0 0	0.39565	0.1	127.376					
7 4 1	0.38961	0.5	131.091					
8 1 1	0.38961	0.3	131.091					
5 5 4	0.38961	0.3	131.091					
8 2 0	0.38384	0.3	135.029					
6 4 4	0.38384	0.3	135.029					
6 5 3	0.37832	0.6	139.257					
8 2 2	0.37303	0.3	143.877					
6 6 0	0.37303	0.1	143.877					
7 4 3	0.36795	0.6	149.106					
7 5 0	0.36795	0.3	149.106					
8 3 1	0.36795	0.6	149.106					
6 6 2	0.36308	0.4	155.271					
7 5 2	0.35839	1.1	163.450					

Table 5.2.10.6. *Fluorophlogopite 00l standard reflection angles [NIST SRM 675, d(001) = 9.98104 (7) Å, T = 298 K, λ = 1.5405929 Å]*

l	2θ (°)
1	8.853
2	17.759
3	26.774
4	35.962
5	45.397
6	55.169
7	65.399
8	76.255
10	101.025
11	116.193
12	135.674

Table 5.2.10.7. *Silver behenate 00l standard reflection angles [d(001) = 58.380 (3) Å, λ = 1.5405929 Å (Huang, Toraya, Blanton & Wu, 1993)]*

l	2θ (°)
1	1.512
2	3.024
3	4.537
4	6.051
5	7.565
6	9.081
7	10.599
8	12.118
9	13.640
10	15.164
11	16.691
12	18.221
13	19.754

The forward reflections have been used in parallel-beam synchrotron-radiation lattice-parameter studies (Parrish *et al.*, 1987).

(3) The profile shape has a strong influence on the accuracy of the angle measurement. The geometrical aberrations produce asymmetries that reduce the accuracy; the effects can be minimized by a proper selection of slit sizes. In most cases, it is inadvisable to use $K\beta$ radiation to avoid $K\alpha$-doublet splitting, as the intensity is reduced by a factor of seven. Symmetrical profiles are obtained with parallel-beam optics, but it is usually necessary to use synchrotron radiation to achieve sufficient intensity.

(4) The largest and commonest source of systematic error in focusing geometry is the specimen-surface displacement. Several remountings of the specimen in the diffractometer and measurement of some low-angle reflections may be helpful in determining and minimizing the error. This aberration does not occur in parallel-beam geometry unless a receiving slit is used.

(5) The precision of the diffractometer gears (or the equivalent) may be the limiting factor in high-precision measurements. The use of an electromagnetic encoder mounted on the 2θ-output shaft can increase the precision considerably. It is not normally included in commercial diffractometers because of its cost, but it is essential for adequate accuracy when the 2θ angles must be determined to better than 0.001°. The various types of mechanical error have been described by Jenkins & Schreiner (1986).

The diffractometer must be carefully adjusted to avoid mechanical problems. The effect of backlash can be minimized by slewing beyond and then returning to the starting angle, and by always scanning in the same direction. It is essential to avoid over-tight worm-and-gear meshing, as it causes jerky rather than smooth movement.

(6) The beam must be precisely centred, the slits and monochromator (if used) must be parallel to the line focus of the X-ray tube, and the scanning plane must be perpendicular to the line focus.

(7) The use of standard specimens with accurately known lattice parameters (Section 5.2.10) and ideally free of line broadening is strongly recommended as a test of the overall precision of the instrumentation and method.

(8) For a given total time available for an experiment, it is necessary to strike a balance between numerous short steps with short counting times and fewer longer steps with longer counting times. The former alternative may give a better definition of the line shape; the latter may give lower calculated standard uncertainties (formerly called estimated standard deviations) in any derived parameters. Obviously, the step length must be considerably shorter than the width of any feature of the profile that is considered to be of importance.

(9) Least-squares refinement is discussed in Subsection 5.2.3.2. The programs and the methods of handling the data should be carefully checked, as various programs have been found to give slightly different values from the same experimental data (see, for example, JCPDS – International Centre for Diffraction Data, 1986; Kelly, 1988).

(10) Specimen preparation is very important; the particle size should preferably be less than 10 μm, and a flat smooth surface normal to the diffraction vector is essential. The linearity of the detector and the temperature of the

Table 5.2.11.1. *NIST intensity standards, SRM 674*

Standard	Crystal system	a_0 (Å)	c_0 (Å)	I_{rel} hkl 2	I_{rel} hkl 3	$I_1/I_c(113)$
Al_2O_3 (corundum)	Trigonal	4.75893 (10)	12.9917 (7)	92.5 (26) 116	87.4 (19) 104	—
ZnO	Hexagonal	3.24981 (12)	5.20653 (13)	57.6 (11) 100	40.2 (14) 002	5.17 (13) 101
TiO_2 (rutile)	Tetragonal	4.59365 (10)	2.95874 (8)	56.9 (28) 211	44.0 (17) 101	3.39 (12) 110
Cr_2O_3	Trigonal	4.95916 (12)	13.5972 (6)	94.5 (22) 116	87.1 (23) 110	2.10 (5) 104
CeO_2	Cubic	5.41129 (8)	—	53.5 (20) 220	43.4 (23) 311	7.5 (2) 111

specimen must be properly controlled during the collection of the experimental data.

(11) The accuracy of the 2θ measurements is directly dependent on the individual step-scanned points. The counting statistical accuracy is determined by the intensity and the background level, and is a major factor in lattice-parameter precision. Preliminary tests on typical profiles ensure that fullest advantage can be taken of the experimental conditions.

(12) At present (2003), the best approach to precision lattice-parameter determination is to follow the suggestions listed above, and to use peak search or profile fitting to calculate the observed 2θ positions. All the well determined peaks are used in the least-squares refinement against 2θ to obtain the zero-angle calibration correction, and in the case of focusing methods the specimen-surface displacement is added. The use of standards is recommended.

references