International Tables for Crystallography (2018). Vol. H, Figure 3.1.25, p. 238.

Table 3.1.2
Run-time parameters used for collection of the data used for certification of SRM 660b

The 'overhead time' associated with the operation of the goniometer is included.

hkl	Start angle (°)	End angle (°)	Step width (°)	Count time (s)	Total peak time (min)
100	20.3	22.2	0.01	2	6.3
110	29.1	31.4	0.01	1	3.8
111	36.4	38.4	0.01	3	10.0
200	42.7	44.4	0.01	5	14.2
210	48	50	0.008	2	8.3
211	53.2	54.896	0.008	5	17.7
110	62.5	64.204	0.008	11	39.0
300	66.7	68.596	0.008	4	15.8
310	70.9	72.7	0.008	6	22.5
311	75	76.904	0.008	9	35.7
222	79.3	80.804	0.008	47	147.3
320	83	84.904	0.008	15	59.5
321	86.9	88.9	0.008	8	33.3
400	95	96.704	0.008	42	149.1
410	98.6	100.8	0.008	9	41.3
330	102.7	104.9	0.008	12	55.0
331	106.9	108.9	0.01	27	90.0
420	111.1	113.1	0.01	20	66.7
421	115.3	117.6	0.01	10	38.3
332	119.9	122.1	0.01	19	69.7
422	129.6	131.796	0.012	32	97.6
500	134.9	137.396	0.012	27	93.6
510	140.5	144	0.014	7	29.2
511	147.5	150.908	0.016	15	53.2
					Total time = 20.0 hours

second involved a simple continuous scan of fixed step width and count time. It is generally accepted that a step width should be chosen so as to collect a minimum of five data points above the full-width-at-half-maximum (FWHM) to obtain data of sufficient quality for a Rietveld analysis (Rietveld, 1967, 1969; McCusker *et al.*, 1999). This does not, however, constitute any sort of threshold; collecting data of a finer step width can, with proper data analysis, result in a superior characterization of the IPF. However, one must consider the angular range of acceptance of the receiving slit that is chosen. For a slit of 0.05° a step width of 0.005° would add only 10% 'new' information, so selecting this step width would not be worth the extra data-collection time. We did, however, collect some data sets we refer to as 'ultra-high-quality' data; the step widths for these were half those shown in Table 3.1.2 and the count times were approximately three times higher than those in Table 3.1.2. For the reported instrument and configuration, the run-time parameters of Table 3.1.2 result in a minimum of 8 to 10 points above the FWHM. Count times were selected to obtain a uniform number of counts for each profile. It should be noted that it is probably not worth spending time collecting quality data from the 222 line of LaB_6, as it is of low intensity and relatively close to other lines of higher intensity; however, this is not the case with the 400 line. Selection of the run-time parameters can be an iterative process; the total width of each profile scan was set to include at least 0.3° 2θ of apparent background on either side of the profile. Except for the data for SRM 676a, the continuous scans discussed were collected with a step width of 0.008° 2θ and a count time of 4 s to result in a scan time of roughly 24 h. The scans of 676a were collected with 0.01° 2θ step width and 5 s count time.

The PSD used on the NIST diffractometer was a one-dimensional silicon strip detector operated in picture-taking mode for all data collection. It has an active window length of 14.4 mm that is divided into 192 strips for a resolution of 75 µm. With a goniometer radius of 217.5 mm this constitutes an active angular range of 3.80° with 0.020° per strip. Slits that would limit the angular range of the PSD window were not used; with each step the counts from all 192 channels were recorded. The PSD was stepped at 0.005° 2θ, for 25% new information per strip; however, to reduce the data-collection time a second coarse step was also included. Therefore, the data-collection algorithm includes the selection of three parameters: a fine step of 0.005°, the number of fine steps between coarse steps (4), and the size of a coarse step (typically 0.1° or 0.2° 2θ). This approach allows for the collection of high-resolution data without stepping through the entire pattern at the high-resolution setting. Data were collected with four fine steps per detector pixel and a coarse step of 0.1° 2θ. They were processed to generate *x*–*y* data for subsequent analysis. The operator can select the portion of the 192 channels, centred in the detector window, to be included in the generation of the *x*–*y* file. The PSD was fitted with a 1.5° Soller slit for collection of the data presented here.

3.1.5. Data-analysis methods

Data-analysis procedures can range from the entirely non-physical, using arbitrary analytical functions that have been observed to yield reasonable fits to the observation, to those that exclusively use model functions, derived to specifically represent the effect of some physical aspect of the experiment. The non-physical methods serve to parameterize the performance of the instrument in a descriptive manner. The origins of two of the most common measures of instrument performance are illustrated in Fig. 3.1.25. The first is the difference between the apparent position, in 2θ, of the profile maximum and the position of the Bragg reflection computed from the certified lattice parameter. These data are plotted *versus* 2θ to yield a $\Delta(2\theta)$ curve; a typical example is shown in Fig. 3.1.26. An illustration of the half-width-at-half-maximum (HWHM), which is defined as the width of either the right or left half of the profile at one half the value of maximum intensity after background subtraction, is also shown in Fig. 3.1.25. These values can be summed to yield the FWHM, and plotted *versus* 2θ to yield an indication of the profile breadth as it varies with 2θ (Fig. 3.1.27). In addition, the left and right HWHM values of Fig. 3.1.28 gauge the variation of profile asymmetry with 2θ; additional parameters of interest, such as the degree of Lorentzian and Gaussian contribution to profile shape,

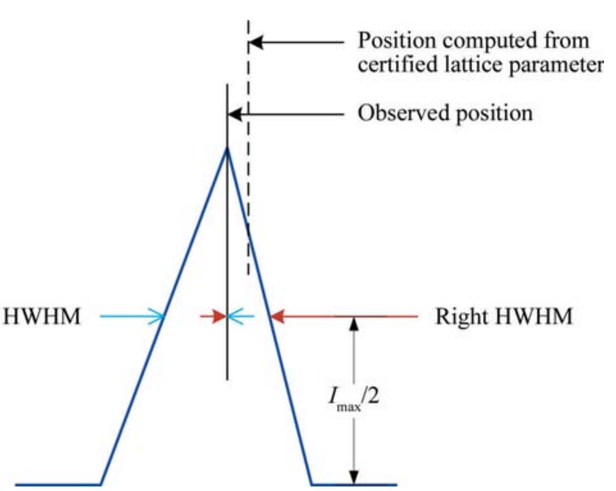

Figure 3.1.25
Diagrammatic representation of a powder-diffraction line profile, illustrating the metrics $\Delta(2\theta)$ and half-width-at-half-maximum (HWHM). The full-width-at-half-maximum (FWHM) = left HWHM + right HWHM.

references