International Tables for Crystallography (2018). Vol. H, Figure 3.1.30, p. 242.

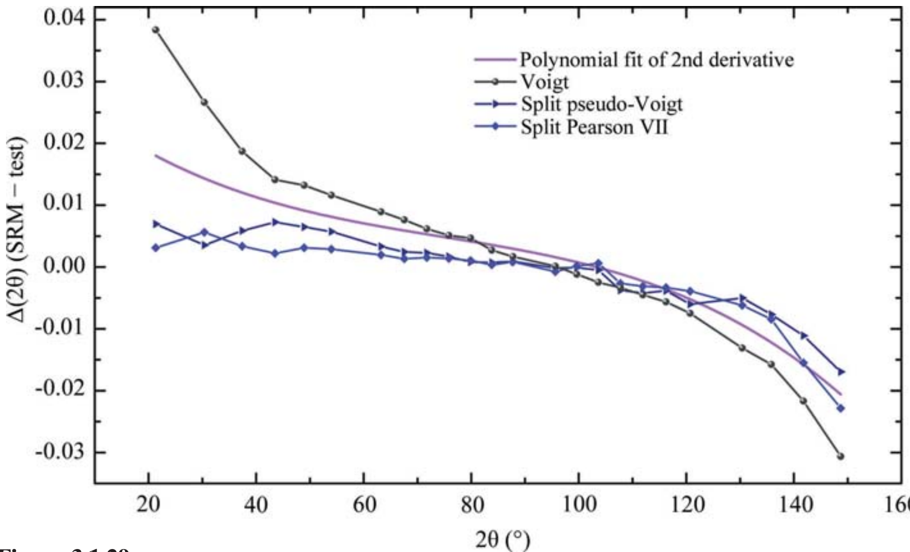

Figure 3.1.29
Comparison of $\Delta(2\theta)$ curves determined with profile fitting of SRM 660b data without the use of any constraints, as a function of 2θ.

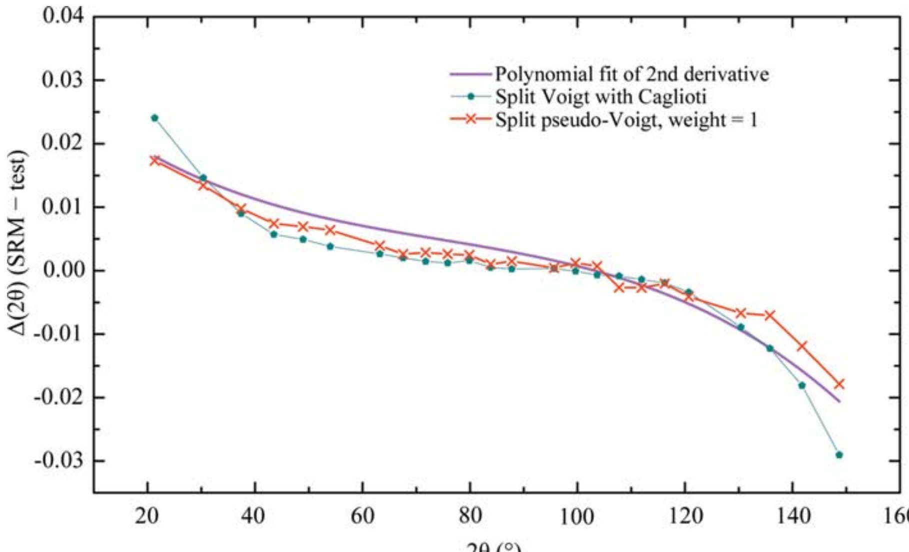

Figure 3.1.30
$\Delta(2\theta)$ curves from SRM 660b determined with profile fitting using the Caglioti function and the unconstrained split pseudo-Voigt PSF with uniform weighting.

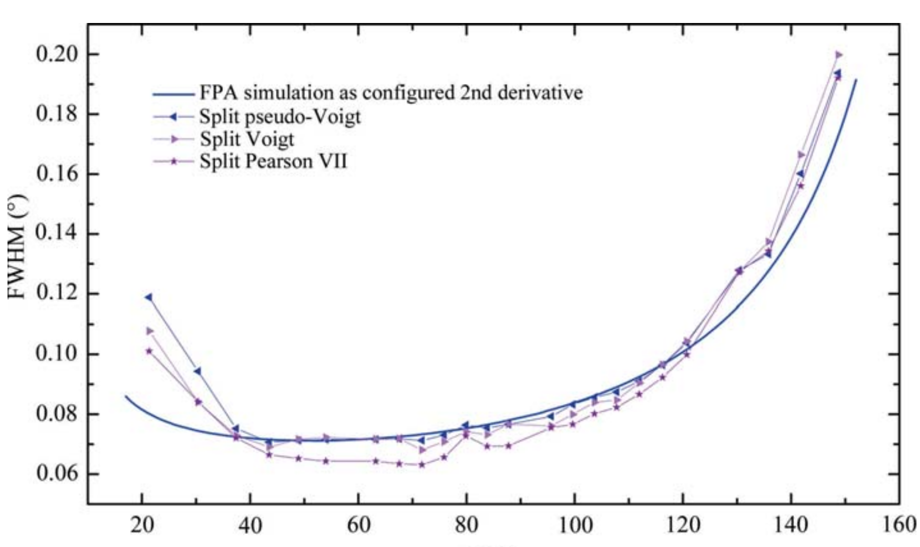

Figure 3.1.31
FWHM data from SRM 660b using various split PSFs fitted without constraints.

generate data for successful analysis with the FPA method, *i.e.* the metrological loop is closed. At low 2θ the profiles are displaced to low angle by the effects of the flat specimen error and axial divergence. The $\Delta(2\theta)$ curve crosses the zero point at approximately 100° 2θ where the profiles are largely symmetric; the slight asymmetry to low angle caused by the flat specimen error is somewhat offset by asymmetry of the emission spectrum at high angles. At higher 2θ the profiles are displaced to high angle by the combined effects of axial divergence and the asymmetry of the emission spectrum. As illustrated with the simulations at lower and higher resolution, the experimental curve of Fig. 3.1.26 would either flatten out or become steeper, respectively, with changes in instrument resolution. Given the uniformity of the data and overall plausibility of this $\Delta(2\theta)$ curve, the third-order polynomial fit is used as a reference against which the merits of other techniques can be judged.

It should also be noted that the data and method shown Fig. 3.1.26 constitute the 'low-hanging fruit' of powder diffraction. Data analogous to those of Fig. 3.1.26 can be used to correct peak positions of unknowns *via* either the internal- or external-standard method using a polynomial fit. The external-standard method, however, cannot account for specimen displacement or sample-transparency effects; these require use of the internal-standard method, which is the same procedure but applied to a standard admixed with the unknown. Either of these methods will correct for instrumental aberrations regardless of their form; the nature of the curve of Fig. 3.1.26 need only be continuous to permit modelling with a low-order polynomial. Studies performed in conjunction with the International Centre for Diffraction Data (ICDD) demonstrate that the use of the internal-standard method routinely yields results that are accurate to parts in 10^4 (Edmonds *et al.*, 1986). Fawcett *et al.* (2004) demonstrated the direct relationship between the use of standards, with the vast majority of analyses being performed *via* the internal- or external-standard methods, and the number of high-quality starred patterns in the ICDD database. Thus, the community's collective ability to perform the most routine of XRPD analyses, qualitative analysis, has been greatly enhanced over the past 30 or so years by these most basic methods and the use of SRMs.

The $\Delta(2\theta)$ and FWHM calibration curves shown in Figs. 3.1.27–3.1.31 were determined *via* profile fitting, using several PSFs,

references