X-STRIPPING with ME: An improved numerical method for deriving monochromatic datasets from laboratory polychromatic diffractograms based on Maximum Entropy.

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Stripping X-ray powder diffractograms from spurious overtones [e.g., K\(\alpha_2\), K\(\beta\) lines] has been around for more than fifty years, since the pioneering work by WA Rachinger (J. Sci. Instrum. 25:254, 1948; Current Contents 42, 1983). Additional successful algorithms along these lines have been published more recently by Ladell et al. (J. Appl. Cryst. 8:499, 1975) followed by Dong et al. (J. Appl. Cryst. 32:168, 1999) and by Ida & Toraya (J. Appl. Cryst. 35:58, 2002). More salient aspects are further detailed in the review by Langford & Louër (Rep. Prog. Phys. 59:131, 1996). The stake is enormous since it results in the ability to index even more complex powder diffractograms using the ubiquitous workhorses DICVOL, NTREOR & ITO and possibly solve the associated crystallographic structures.

Our new method is quite general and may involve more than one or two overtones to be suppressed. It presents itself as a two-step procedure. During the first, one reference sample (e.g., SRM 676 [corundum]) is carefully measured and then Rietveld-refined yielding the 3 [U,V,W] Caglioti parameters describing the lineshape dependence versus the scattering angle \(\theta\) for each one of the involved wavelengths. The point spread function (PSF) is then computed, which features as many Gaussian peaks as wavelengths. In a second step, Maximum Entropy (ME) is use to unfold the measured diffractogram from the PSF and the resulting unfolded spectrum is folded again with the lineshape pertaining to the uniquely wanted wavelength (i.e., K\(\alpha_1\)) to produce a monochromatic X-stripped dataset. A side benefit resulting from this last folding is its noise-suppressing effect.

Our current default FORTRAN implementation of this strategy is called X-STRIPPER. It features the removal of both K\(\alpha_2\) and K\(\beta\) and aims at producing quasi-monochromatic K\(\alpha_1\) diffractograms. It also makes use of the Caglioti parameters [U,V,W] as defined in the GSAS XRPD package. In practice, the latter triplets are taken to be identical for the fundamental wavelength K\(\alpha_1\) and the remaining overtones even though they need not be. Our code runs within a few minutes on a PC laptop for diffractograms up to 9000 data points. We have successfully tested it using raw datasets obtained from our laboratory [Bruker AXS] D8 Advance diffractometer using a Bragg-Brentano geometry. Quite a few minerals [corundum, SiC, gypsum, aragonite …] and one organic compound [Sorafenib Tosylate] with lattice symmetries ranging from hexagonal down to monoclinic were investigated and their respective unit cells were always found using either DICVOL06 and/or N-TREOR09. In the case of organics, the use of an appropriate sample-holder is mandatory to reduce spurious diffuse scattering and transparency effects to a minimum. Indeed, our entropic strategy may fail in the presence of the latter.

The Authors are hugely indebted to Prof. Daniel Louër for help and advice associated with the use of his Indexing Package DICVOL06, as well as for key suggestions regarding their experimental set-up. This work is most respectfully dedicated to him.