

## The Certification of SRM 1979 for Analysis of Crystallite Size

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This rather long-standing NIST SRM project constitutes an effort to provide a standard for the analysis of crystallite size from the consideration of powder diffraction profile broadening. The standard will consist of two ZnO powders, the first with a crystallite size in the 20 nm range and a second with size in the 70 nm range. We report on the various avenues that were investigated for the preparation of the feedstock; these were derivative of the extensive body of work published by D. Louër, *et al.*, on the oxides of cerium and zinc. After considerable effort, and a number of unfortunate setbacks, we proceeded with the ex-oxalate route for the preparation of ZnO feedstock. A specialized vacuum furnace was built in order to decompose the zinc oxalate in a highly-controlled manner. Certification data were collected on a NIST built diffractometer equipped with a Johansson incident beam monochromator, sample spinner and scintillation detector. This machine yielded diffraction profiles that could be fit accurately using analytical profile shape functions and SRM 660b was used to determine the instrument profile function. Data were analyzed using several codes, with TOPAS<sup>1</sup> being used to obtain the certified lattice parameters and PM2K<sup>2</sup> being used to determine microstructural data. These microstructural data are reported as ancillary information. These ZnO powders may be less than ideal as stacking faults, as reported by J.I. Langford, *et al.*<sup>3</sup> are observed. However this is a second-order effect and many lines are not influenced by these defects. The crystallites are in the form of discs of a fairly small aspect ratio. We report on TEM data that largely confirm the XRD results.

- 1) TOPAS, *General Profile and Structure Analysis Software for Powder Diffraction Data*, V4.2, Bruker AXS GmbH, Karlsruhe, Germany
- 2) M. Leoni, T. Confente & P. Scardi, *PM2K: a flexible program implementing Whole Powder Pattern Modeling*, *Z. Kristallogr. Suppl.* 23 (2006) 249-254
- 3) J.I. Langford, A. Boultif, J.P. Auffrédic and D. Louër, *The Use of Pattern Decomposition to Study the Combined X-ray Diffraction Effects of Crystallite Size and Stacking Faults in Ex-Oxalate Zinc Oxide*, *J. Appl. Cryst.* (1993) 26, 22-33