

Development of standard reference materials for powder diffraction

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The calibration and alignment of powder diffraction equipments, both neutron and x-rays diffractometers, is generally performed by means of measurement of standard samples. These samples must have very well defined cell parameters and high stability. In experiments using synchrotron radiation it is also necessary a precise determination of the energy (or wavelength) of the radiation, which can be made using a standard sample. For the determination of crystallite sizes and microstrains it is usual to determine the instrumental parameters, both in conventional laboratory and synchrotron diffractometers. In this case, besides having the crystal structure and cell parameters very well defined, it is imperative that the standard sample presents high crystallite size and low (or none) microstrains, in order to obtain diffraction peaks broadening due only to instrumental factors. For this purpose normally are used the “NIST Standard Reference Materials for Powder Diffraction^[1]” like Al₂O₃, Si, LaB₆ etc. In order to have an alternative source and attend the demand of Brazilian XRD research community, we are developing a set of materials that attempt to the requirements to be used as powder diffraction standards. We are working in the production of standard samples of Al₂O₃, Y₂O₃, among others. In this work are presented some preliminary results of these standard samples, measured in conventional equipment and synchrotron (D10B-XPD-LNLS), both in high- and low-resolution configurations. The experimental data were analyzed by Rietveld refinement^[2] and “fundamental parameters method^[3]” in order to determine crystallite size and microstrain, and are compared to the NIST LaB₆ standard. The results show that the samples fulfill the requirements to be used as powder diffraction standards.

Keywords: Powder diffraction, standard reference materials, Rietveld refinement, crystallite size, microstrain.

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[1] NIST, May 14, 2008, <http://ts.nist.gov/measurementservices/referencematerials/index.cfm>

[2] H. M. Rietveld, *J. Appl. Cryst.* 2 (1969) 65-71

[3] A. Coelho, "Topas Academic," 4.1 ed. Brisbane, Australia: Coelho Software, 2007, p. TOPAS ACADEMIC.