COMBINED REFINEMENT OF SYNCHROTRON BaPb<sub>x</sub>Bi<sub>2</sub>O<sub>3</sub> POWDER DATA FROM THE SUPERCONDUCTOR AND NEUTRON POWDER DATA FROM THE SUPERCONDUCTOR 

By H.K. Maiche, J.P. Hringer, J.P. Wroblewski, and A.W. Hewat 

The Rietveld structure refinement technique has, over the past two decades, proved to be the most important tool for the evaluation of neutron and X-ray powder patterns. However, often it is difficult to obtain new data from a single data set, even with an unambiguous structural model. In BaPb<sub>x</sub>Bi<sub>2</sub>O<sub>3</sub> Cox et al. (SAO: State Comm. 19, 959, (1976)) concluded from neutron powder data a tetragonal structure at room temperature, but high resolution X-ray Guinier diffractometer recordings taken at 300 K exhibited a monoclinic distortion (I=90.089° (2)°). Measurements of 19 reflection groups with synchrotron radiation at an high resolution 3-axis powder diffractometer at HASYLAB/DESY, confirmed the monoclinic symmetry. Neutron data collected at instrument DIA at ILL (Grenoble) ( λ=1.27 Å) confirmed the monoclinic symmetry. The data set was refined using the new program JACINTH (J. Appl. Cryst. 15, 430–438 (1982)). The new program improved the fit of the major phases, whose crystal structure was known, were simulated and the pattern indexed.

High resolution powder diffraction, using the ISIS spallation neutron source at the Rutherford Laboratory, UK (M.W. Johnson, W.I.F. David and W.T.A. Harrison, RAL 86–068) has been used to obtain data on SYNROC with no waste, with 10% waste, and with 20% waste. High level waste was simulated by the use of non active isotopes.

The data was first smoothed by the maximum entropy deconvolution algorithm (S. Steenstrup, Aust. J. Phys. 1985, 38, 319). The diffraction patterns of the major phases, whose crystal structure are known, were simulated and the pattern indexed. The data corresponding to each of these phases was stripped from the pattern and Rietveld refinement used to determine the concentration and lattice parameters.

Data from the minor phases was isolated for an attempt to find their structures by direct methods (A.K. Cheetham, W. I. F. David, M. M. Eddy, N. J. B. Jackman, M. W. Johnson and C. C. Toradi (Nature 1986, 320, 46)).