12.5-3 USE OF THE RIETVELD TECHNIQUE FOR DETERMINING CATION DISTRIBUTIONS. By Anders G. Nord and Theodor Stefanidis*, Section of Mineralogy, Swedish Museum of Natural History, Box 50007, S-104 05 Stockholm, Sweden.

The Rietveld full-profile refinement technique (J. Appl. Cryst. 2, 1969, 65-71) has been used to determine cation distributions among Me** ions in numerous solid solutions. These investigations have been based on neutron powder diffraction data or photographic X-ray Guinier-Hägg data (cf. Malmros & Thomas, J. Appl. Cryst. 10, 1977, 7-11). The mineral and inorganic oxosalt structures studied are comparatively simple. The refinements include atomic parameters, a scale factor, peak-profile parameters, and unit cell dimensions, which implies 14 parameters to be refined for olivines, 25-32 parameters for fayalite and saxitolidine, tetratetrahedrites and 84 for goethite, epsomite, and hematite. The CATION DISTRIBUTIONS.

The main routine, the X-ray Rietveld program in an intuitively simple way within the framework of the full-profile refinement was satisfactorily accomplished on the basis of a monoclinic cell, space group P2_1/c, with refined lattice constants a = 6.10390(6) Å, b = 6.03392(6) Å, c = 9.53614(15) Å, β = 90.4325 (15) deg. A striking feature of this refinement is that the angular variation of the peak halfwidths and the relative contributions of Gaussian and Lorentzian components is described satisfactorily by only two variable parameters, one for the instrumental resolution and the other for the particle size broadening. The structure may be viewed as a modification of the pseudo-cubic approximation (Winchell, Butler, West, and Buchanan, Rev. Sci. Instr. 1974 45, 1369; Young and Wiles, J. Appl. Cryst. 1982 15, 430). With these modifications, Rietveld refinement was satisfactorily accomplished on the basis of a monoclinic cell, space group P2_1/m, with refined lattice constants a = 6.10390(6) Å, b = 6.03392(6) Å, c = 9.53614(15) Å, β = 90.4325 (15) deg. A striking feature of this refinement is that the angular variation of the peak halfwidths and the relative contributions of Gaussian and Lorentzian components is described satisfactorily by only two variable parameters, one for the instrumental resolution and the other for the particle size broadening.

The structure may be viewed as a modification of an ideal ordered perovskite-type structure with Fm3m symmetry in which there is cooperative tilting of rigid SrO layers and distorted six-membered rings of oxygen atoms, a pseudo-cubic [110] axis, very similar to that of Ba2Fe12O19 (Cox and Slegten, Solid State Commun. 1976 19, 969; Thornton and Jacobson, Acta Cryst. 1978 B34, 351). Neutron data collected from the same sample confirm this structure.

This work is supported by the U.S. Department of Energy under contract DE-AC02-76CH00016.

12.5-4 THE X-RAY RIETVELD SYSTEM XRS-84. By Ch. Baerlocher, Institut für Kristallographie und Petrographie, ETH, Zürich, Switzerland.

The X-ray Rietveld System is a collection of programs designed to carry out the calculations required to solve and refine crystal structures with the Rietveld technique using x-ray or neutron powder data. It has been successfully used in a number of cases, especially for the refinement of complex zeolite structures (e.g. TPA-ZSM-5 with 181 structural parameters (BAERLOCHER, Proc. 6th Int. Conf. Zeolite Conf. 1983, 245-269, Butterworth, UK).)

XRS-84 is an extended and perfected version of XRS-82 and is based on the X-ray System (Stewart et al., Acta Cryst. B28, 2372-2382, 1972; Stewart, B. E., A. C. modulus and a modified Philips diffractometer equipped with a perfect Ge(111) analyzing crystal. This work is supported by the U.S. Department of Energy under contract DE-AC02-76CH00016.


It has recently been demonstrated that the high resolution of powder diffraction data which can be obtained with synchrotron radiation may have important applications to structure analysis by means of Rietveld refinement (Cox, Hastings, Thomlinson, and Prevette, Nucl. Instr. and Meth. (1983) 208, 373; Hastings, Thomlinson and Cox, J. Appl. Cryst. (1984), in press). This technique has been applied to a refinement of the structure of Ba2SrW06 which was originally reported to be a moderately distorted form of the cubic Ba2CuO3 structure (Steward and Booksby, Acta Cryst. 1951 4, 503), and subsequently to have a monoclinic cell (Hovka, Lykova and Shevchenko, Russ. J. Inorg. Chem. (1971) 16, 1150).

Synchrotron x-ray data were collected at the Cornell High Energy Synchrotron Source (CHESS) with x-rays of wavelength 1.54 Å from a perfect Si(220) double-crystal monochromator and a modified Phillips diffractometer with a perfect Ge(111) analyzing crystal.

The observed peaks were intermediate in shape between Gaussian and Lorentzian, and significantly broadened with respect to the instrumental resolution function, with an angle dependence qualitatively consistent with a predominantly Lorentz-like particle size broadening. The effects of this and also strain broadening were taken into account by modifications to the Rietveld program in an intuitively simple way within the framework of the pseudo-cubic approximation (Hertheim, Butler, West, and Buchanan, Rev. Sci. Instr. 1974 45, 1369; Young and Wiles, J. Appl. Cryst. 1982 15, 430). With these modifications, Rietveld refinement was satisfactorily accomplished on the basis of a monoclinic cell, space group P2_1/m, with refined lattice constants a = 6.10390(6) Å, b = 6.03392(6) Å, c = 9.53614(15) Å, β = 90.4325 (15) deg. A striking feature of this refinement is that the angular variation of the peak halfwidths and the relative contributions of Gaussian and Lorentzian components is described satisfactorily by only two variable parameters, one for the instrumental resolution and the other for the particle size broadening.

The structure may be viewed as a modification of an ideal ordered perovskite-type structure with Fm3m symmetry in which there is cooperative tilting of rigid SrO layers and distorted six-membered rings of oxygen atoms, a pseudo-cubic [110] axis, very similar to that of Ba2Fe12O19 (Cox and Slegten, Solid State Commun. 1976 19, 969; Thornton and Jacobson, Acta Cryst. 1978 B34, 351). Neutron data collected from the same sample confirm this structure.

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