

Programming (1975), 9, 1-30). The latter is less sensitive with respect to high correlation since it uses a positive definite matrix with elements converging towards the second order derivatives of the function. This greatly improves the behavior around ill-defined minima, which are frequently encountered in the Rietveld method (R.A. Young (1980), NBS Special Publication 567, 143-162).

In addition to structural parameters, the program can refine lattice constants, a linear  $2\theta$  correction, peak half width and asymmetry, and preferred orientation. It can also handle the broadening of individual lines due to crystallite shape or lattice strain. The data input is kept to a minimum and is partly in free format. At the moment the program requires strictly monochromatic diffractometer data, which are necessary to achieve good resolution. Neutron data can also be employed.

The programs have been successfully tested in a number of different structure refinements including that of zeolite ZSM-5. This zeolite has an orthorhombic structure with a cell of  $20 \times 20 \times 13 \text{ \AA}^3$  and 134 positional parameters. The X-ray data were supplemented with 165 distance and angle constraints.

**12.5-03** ANALYSIS OF TIME-OF-FLIGHT DATA FROM A RESOLUTION FOCUSED NEUTRON POWDER DIFFRACTOMETER. By R.B. Von Dreele, Department of Chem., Ariz. State Univ., Tempe, Ariz. 85281 and A. Soper, Los Alamos Scientific Laboratory, Los Alamos, N.M.

The resolution focused powder diffractometer at the WNR neutron pulsed source has five counter banks positioned at average  $2\theta$  values of 150, 90, 40, 12.5 and  $7^\circ$  having 16, 16, 6 and 8 detectors each, respectively. The banks are tilted so that all tubes in a bank have the same resolution ( $\Delta d/d$ ) for a given  $d$ -spacing. Because the resolution focusing condition is not the same as the time focusing condition for a given counter bank, the spectra associated with the counters in a bank do not coincide in time and must be recorded separately.

The methods used to reduce the 62 individual spectra to a more manageable number and some results from Rietveld refinement of the merged data from some test samples will be presented.

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**12.5-04** USE OF THE COMPUTER CONTROLLED POWDER DIFFRACTOMETER FOR THE CRYSTAL STRUCTURE DETERMINATION OF SEVERAL RARE EARTH MAGNESIUM FLUORIDES. By R. Jenkins, and E. Banks, Polytechnic Institute of New York, Brooklyn, NY.

This paper describes the use of the computer controlled powder diffractometer for the determination of the crystal structures from powder data, using as examples of several Rare Earth Magnesium Fluorides. The lack of specimens of sufficient purity precluded use of profile fitting techniques so use was made of more classical methods of peak location and intensity measurement. An attempt was made to establish the limitations of such a procedure both in the accuracy obtainable in terms of cell parameters and also in the elucidation of atomic arrangement.

Three materials were examined, these being the mixed Magnesium Fluorides of Eu, Sm and Sr. Orthorhombic structures were assigned and the unit-cell dimensions were found to be  $\text{EuMgF}_4$ : 3.395, 14.43 and 5.664 Å; for  $\text{SmMgF}_4$ : 3.695, 14.44 and 5.661 Å and for  $\text{SrMgF}_4$ : 3.917, 14.51 and 5.637 Å. It is estimated that the accuracy of the cell parameter data is of the order of 3 parts per 10,000. Each unit cell had 24 atoms corresponding to 4 formula weights/cell. R-factors of 0.10, 0.17 and 0.18 were found for the three structures respectively. Each Rare Earth and Strontium atom was found to be in 8-fold coordination with the Fluorine atoms and the Magnesium atoms in 6-fold coordination with the Fluorines. The Rare Earth and Strontium atom to 6-fold coordination with the Fluorines. The Rare Earth and Strontium atom to Fluorine distance was found to be 2.32-2.8 Å and the average Magnesium to Fluorine distance 2.03 Å. These distances are in agreement with the values of 2.55 Å and 2.02 Å reported by Shannon.

**12.5-05** X-RAY GUINIER POWDER PROFILE REFINEMENT OF  $\text{Rb}_2\text{NaHoF}_6$  AT 17 K.

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$\text{Rb}_2\text{NaHoF}_6$  (Fm3m,  $a = 8.8622(2) \text{ \AA}$  at 205 K) exhibits a  $\Gamma$ -point phase transition at  $170 \pm 5 \text{ K}$  with a continuously increasing tetragonal distortion. The diffraction pattern ( $5^\circ < \theta < 46.2^\circ$ ) at 17 K was recorded with an automated low temperature Guinier diffractometer (Ihringer, J. of Appl. Cryst. (1980) in press). For the structure refinement the modified Rietveld program (Young, Mackie & von Dreele, J. Appl. Cryst. (1977) 10 262) with correction factors appropriate to the Guinier geometry was used.

All models resulting from a one phonon  $\Gamma$ -point condensation were considered. The best final residual of 4.6 % was found for the space group  $I4/m$  ( $T=17 \text{ K}$ ,  $a=6.2233(2)$ ,  $c=8.8957(3) \text{ \AA}$ ,  $V=344.53 \text{ \AA}^3$ ). The  $\text{NaF}_6$  octahedra remain undistorted, but they are tilted around the (001) axes within an angle of 5.45 deg. This kind of distortion is well known from the phase transitions in perovskites or  $\text{K}_2\text{PtCl}_6$  structures. In  $\text{Rb}_2\text{NaHoF}_6$ , however, the tetragonal distortion - a sterical result of the tilted octahedra - is being developed without any measurable discontinuity ( $\Delta a, c \leq 0.007 \text{ \AA}$ ), and increases almost linearly with decreasing temperatures. The mechanisms of the lattice stability below the phase transition will be discussed.