12.5-11 THE RIETVELD METHOD - AN ALTERNATIVE APPROACH. By G.B. Mitra and Prabal Das Gupta, CSS Department, Indian Association for the Cultivation of Science, Jadavpur, Calcutta - 700 032, India.

The Rietveld method (H.M. Rietveld, Acta Cryst., 1967, 22, 151-153, H.M. Rietveld, J. Appl. Cryst., 1969, 2, 65-71) of structure refinement is based on the assumption that all peaks in a pattern from a sample can be expressed in terms of a single mathematical function. In the same method full width at half intensity maxima (H_b) is given by $H_b = U\tan^2\theta + V\tan\theta + W$ where θ is the Bragg angle and UVW are constants for sample used. From R(x) test and β_2 test (G.B. Mitra, Acta Cryst., 1963, 16, 429; G.B. Mitra, Brit. J. Appl. Phys., 1965, 16, 77) of peaks in the diffractogram of certain metals and alloys, it has been revealed that all peaks from a particular sample do not always conform to any particular mathematical function. Residuals obtained comparing experimental data of H and those obtained from least square fit is often quite high. Because of these discrepancies an alternative method has been introduced. This new method does not assign any apriori mathematical function to line shapes. It is based upon the fact that nth order Fourier transform of a composite profile is sum of the nth order Fourier transform of constituent peaks. It has also made use of the fact that particle size measured along (h,k,l) direction is also independent of the order of the reflection. Using this method present authors have refined the structure of WO from the data taken from Loopstra et al. (Acta Cryst., 1966, 21, 158-162) and the residual diminshed by 40%.

12.5-12 THE CRYSTAL STRUCTURE OF TRICLINIC SF_6 FROM NEUTRON POWDER DIFFRACTION MEASUREMENTS. B.M Powell, AECL, Chalk River, Ontario, Canada, M.T. Dove, University of Cambridge, G.S. Pawley, University of Edinburgh and L.S. Bartell, University of Michigan, Ann Arbor.

The crystal structure of the low temperature phase of sulphur hexafluoride (SF_6) below 96K has been solved from neutron powder diffraction measurements. Diffraction profiles were measured at the NRU reactor, Chalk River. The application of exhaustive search methods failed to find a unit cell, suggesting that the structure might be triclinic at all temperatures. Molecular dynamics simulations (MDS) and electron diffraction measurements suggested the existence of a hexagonal structure just below 96K with a lower symmetry structure at lower temperatures. Profile refinements with the configurations calculated by MDS as initial values proved unsuccessful. A new procedure, developed to index the powder patterns of low symmetry structures, was applied to the neutron diffraction data. This procedure generates a trial structure from molecular packing considerations and avoids entrapment in false minima by broadening the observed and calculated powder peaks in a controlled manner. It succeeded in indexing the diffraction profile in terms of a triclinic unit cell. With these lattice parameters, with initial molecular positions and orientations from the MDS calculations and assuming space group P1 with Z = 3, the diffraction data were fully refined. The crystal structure is triclinic at 23K and at 85K and no evidence was found for the existence of a hexagonal phase. This is the lowest symmetry structure yet solved from powder diffraction data. The phase transition is interpreted in terms of two separate lattice distortions from the high temperature, cubic phase which couple to different stages of orientational ordering. The mechanism driving the transition is the resolution of orientational frustration as the temperature is lowered through the transition.

12.5-13 RIETVELD ANALYSIS OF POWDER PATTERNS OBTAINED BY TOF NEUTRON DIFFRACTION USING COLD NEUTRON SOURCES. By F. Izumi, National Institute for Research in Inorganic Materials, Sakura-mura, Ibaraki 305, Japan, H. Asano, Institute of Materials Science, University of Tsukuba, Sakura-mura, Ibaraki 305, Japan, and N. Watanabe, National Laboratory for High Energy Physics, Oho-machi, Ibaraki 305, Japan.

A high-resolution time-of-flight (TOF) powder diffractometer, HRP, was constructed at the pulsed spallation neutron facility (KENS) at National Laboratory for High Energy Physics (Watanabe, N., Asano, H., Iwasa, H., Sato, S., Murata, H., Karahashi, K., Tomiyoshi, S., Izumi, F. & Inoue, K. (1987) Jpn. J. Appl. Phys. in press). The instrument utilizes narrow pulsed beams of thermal and epithermal neutrons from a solid methane moderator at 20 K, and a high resolution of $\Delta d/d \sim 0.003$ has been realized at $d < 150$ pm. The present work was undertaken to obtain reliable structure parameters from TOF neutron powder diffraction patterns taken on the HRP by Rietveld analysis. A computer software package, RIETAN, developed for the Rietveld analysis of angle-dispersive powder data has now been modified extensively to make it possible to analyze TOF neutron diffraction data. The program has a number of convenient features, e.g., (1) data bases storing information on space groups, scattering lengths, and cross sections; (2) data input in free format; (3) single-pass operation; (4) multiphase capability; (5) availability of three different methods of least squares: Gauss-Newton, modified Marquardt, and conjugate direction methods; (6) automatic successive refinements; (7) minimization under equal and unequal constraints. A profile shape function is implemented which is the sum of two asymmetric functions (Cole, I. & Windsor, C.G. (1980). Nucl. Instrum. Methods, 171, 107-113) in a (1-R):R ratio. This function, which contains ten variable parameters, can be used without any modifications even if intrinsic broadening originating from the particle size or the strain of a sample is observed. The results of Rietveld refinements of silicon and α-alumina showed that the profile shape function fits neutron diffraction patterns taken on the HRP very well and that highly precise structural parameters can be obtained with this program.