12.X-01 STRUCTURE REFINEMENT FROM POWDER DIFFRACTION DATA - A REVIEW. By A.W. Hewat, Institut Max von Laue-Paul Langevin, BP 156X, 38042 Grenoble, France.

"All the spectra of the different planes are thrown together on the same diagram, and must be disentangled. This is not so difficult as it might seem..." (W.H. Bragg, 1921, Presidential address, Proc. Phys. Soc. 34, 33).

In an age without computers, the difficulties were nonethe-less such that academic crystallographers preferred instead idealised single crystals, leaving the messy world of powders to industrial workers, who usually did not have the choice of samples. However, just as computers have provided completely automatic solutions for single crystal structures, they have, after half a century, permitted powder patterns to be completely disentangled. The various steps in this procedure will be reviewed. (References are to reviews where-ever possible).

1) Profile fitting of the positions of the individual lines (e.g. Parrish & Huang (1980) NBS Special pub. 567 95-110).
2) Automatic indexing of the line positions to obtain the unit cell (e.g. Shirley (1980) NBS cit. 361-382)
3) Profile refinement of the cell parameters and line intensities (e.g. Cooper, Sakata & Rouse (1980) NBS cit. 167-187 ; Pawley (1981) in press)
4) Model building using these cells and space groups with known bond lengths and angles (e.g. Baerlocher, Hepp & Meir (1979) DLS-76 manual, & (1980) NBS cit. 165)
5) Rietveld profile refinement of the model structure (e.g. Hewat (1980) NBS cit. 111-141 ; Young (1980) NBS cit. 143-163 ; Malmros & Thomas (1977) J. Appl. Cryst. 10, 7-11 ; Khattak & Cox (1977) J. Appl. Cryst. 10, 404-411 ; Von Dreele,Jorgensen & Windsor (1981) in press; Sabine (1980) NBS cit. 21-32).
6) Constrained profile refinement of the model (Pawley (1980) J. Appl. Cryst. 13, 630-633)

At each step, information is extracted, with the final objective being to explain the complete profile with the simplest possible model and the least number of parameters. This method, again according to W.H. Bragg (1921) loc. cit.

"promises to be of great use, since every crystal has its own characteristic spectrum".

12.X-02 CRYSTAL STRUCTURE REFINEMENT FROM PROFILE FITTED POWDER DATA. By G.Will, University of Bonn, BONN, West Germany.

The analysis and refinement of a structure with profile fitting methods is performed in two steps: First, GAUSSIAN or LORENTZIAN functions, depending on the type of experiment, are fitted to each observed peak or multiplet. This requires knowledge of the instrument function and its dependence on scattering angle. In the X-Ray case the profiles are asymmetric, and the correct profile function is determined experimentally (Parrish, Huang & Ayers, Trans. Am. Cryst. Assoc.(1976)12,55-73). In neutron and energy dispersive X-Ray diffraction diagrams the profiles are broad Gaussians. This approach provides the basis for making precise measurements of integrated intensities from powder samples. The fitting procedure does not interfere or correlate with the structural parameters in the ensuing refinement.

In the second step these results are used in POWder Least Squares calculations (POWLS) to determine refined positional parameters. This program allows one to consider overlap of multiple peaks (like cubic 333/511) or of peaks not resolvable by profile analysis. The differences and advantages compared to the widely used alternative total pattern fitting procedures, especially in the case of X-Rays with non-Gaussian profiles, are obvious.

The method has been successfully applied for many years in neutron diffraction. Results using these methods with angle dispersive X-Ray diffractometry, precise powder data and profile fiting obtained recently in collaboration with W.Parrish and T.C.Huang at IBM Research Laboratory, San Jose, wil be given (see accompanying abstract by W.Parrish & T.C.Huang).

12.X-03 COMPARISON OF THE RIETVELD METHOD BY SEVERAL DIFFERENT EXPERIMENTAL TECHNIQUES. By B.T.M. Willis, Materials Physics Division, AERE Harwell, England.

In the conventional method of carrying out experiments in neutron and X-ray powder diffraction, a fixed wave-length λ is chosen for the incident radiation and the Bragg peaks are measured by observing the intensity of the scattered radiation as a function of the scattering angle 2θ. An alternative way of satisfying Bragg's equation is to keep θ fixed and to determine the scattered intensity as a function of λ. This second method has been achieved in neutron diffraction by using a pulsed source and time-of-flight analysis of the scattered neutrons. In the corresponding X-ray experiment, the energy-dispersive technique has been employed to measure Bragg intensities at a fixed θ and varying λ. Thus there are four powder diffraction methods, with neutrons or X-rays as the primary radiation, and with the intensity measured at a fixed wavelength or at a fixed scattering angle.

The 'profile refinment' method (Rietveld, J.Appl.Cryst. (1969) 2, 65) was first applied successfully to the interpretation of neutron-diffraction data from polycrystalline samples, but it is now being used more and more widely to interpret X-ray powder patterns. In this paper we shall review the application of the Rietveld method to the analysis of powder diffraction data collected by each of the four experimental procedures mentioned above.