12. X–1 DIRECT METHODS IN CRYSTAL STRUCTURE DETERMINATIONS FROM POWDER DIFFRACTION DATA.
By A. Nørlund Christensen, Department of Chemistry, Aarhus University, DK-8000 Aarhus C, Denmark, and M.S. Lehmann, Institut Max von Laue - Paul Langevin, F-38042 Grenoble Cedex, France.

The majority of structure analysis done with powder data starts from a partially known structure. It is, however, possible to start from a totally unknown structure (A.W. Hewat, The Neutron and its Application, 50th Anniversary Discovery of the Neutron, Cambridge 1982, private communication).

The analysis consists of a series of steps identical to conventional single crystal analysis:
1) Data collection of powder patterns using either neutrons or X-rays (possibly synchrotron radiation). It is essential to aim at extremely high resolution so that at least a limited number of individual reflections are resolved. Information from Guinier photographs may be used as well.
2) Indexing of the powder pattern. Several computer programs are available for this task.
3) Careful extraction of intensities for individual reflections. This can be done by least-squares technique using the known unit cell (G.S. Pawley, see previous talk).
4) Conventional structure analysis using direct methods or Patterson methods, possibly supported by energy calculations or distance least-squares calculations.

Results and difficulties will be discussed for data sets ($Zr(NaPO_4)(DPO_4)D_2O$, $Ca_5(SiO_4)_2(OD)_2$) collected on neutron diffraction instruments, and a comparison with possible identical techniques using X-ray synchrotron radiation will be given.

12. X–2 HIGH RESOLUTION NEUTRON POWDER DIFFRACTION AND RIETVELD REFINEMENT. By J.C. Taylor, Energy Chemistry Division, C.S.I.R.O., Lucas Heights Research Laboratories, P.M.B.7, Sutherland, N.S.W., 2232, Australia.

Rietveld profile analysis with neutron powder data of moderate resolution has been found in our laboratory sufficient to prove new structural types in actinide and transition metal halide and oxyhalide systems. This field will be briefly discussed. More complicated structures require high resolution (HRD) powder data, and an 8-counter HRD became operational at Lucas Heights in 1983. There is now some interest in the limits to which the HRD technique can be pushed before meaningful structural information is lost. Systems demanding HRD data are the numerous sheet, cage and channel structures, where the framework diffracts strongly, but the interstitial atoms (which are the ones of more interest) diffract weakly, being loosely bound. Examples of this are the sheet U phosphate minerals and zeolitic structures. As our Division is now interested in zeolitic compounds because of their catalytic and other uses, we have studied neutron patterns of substituted Y-zeolites, and natural stellerite in various stages of dehydration. These compounds test the limits of the method. Parallel neutron single crystal studies of natural zeolites are presented elsewhere at this Conference (Miller, Hambley, and Taylor, conference abstract). Examples of the application of HRD methods to channel structures, many showing intra-channel disorder, will be described.

12. X–3 STRUCTURAL REFINEMENT FROM PULSED-NEUTRON-SOURCE POWDER DIFFRACTION DATA. By James D. Jorgensen, Argonne National Laboratory, Argonne, IL 60439, USA

The operation of two time-of-flight powder diffractometers at the Intense Pulsed Neutron Source since November 1981 (as well as the operation of previous prototype instruments) has provided considerable experience from which to evaluate the strengths and weaknesses of pulsed-neutron-source powder diffraction. The high resolution obtained in back-scattering has proven very effective for refining structures of moderate complexity. A few refinements of over 100 parameters have been done. The small d-spacing data available at a pulsed source allow atom positions and thermal ellipsoids to be determined to high precision if the cell is not too large. Currently operating diffractometers are less well optimized for large cell structures where data from a smaller scattering angle must be analyzed in order to reach larger d-spacings and avoid serious peak overlap. However, several successful refinements of zeolites and other similarly-sized cells have been done. The fixed angle technique has proven particularly useful for obtaining data for samples in restrictive environments. For example, high pressure data and very low temperature data have been collected at a 90° scattering angle where collimation can completely eliminate scattering from the sample cell. Currently operating pulsed-source diffractometers are competitive with respect to both resolution and overall data rate. Future pulsed neutron sources of higher flux should allow order of magnitude increases in performance.

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12. X–4 CHEMICAL APPLICATIONS OF NEUTRON POWDER DIFFRACTION. By Anthony K. Cheetham, University of Oxford, Chemical Crystallography Laboratory, 9 Parks Road, Oxford, OX1 3PD, U.K.

The applications of powder neutron diffraction to a range of chemical problems will be illustrated with examples using both constant wavelength and time-of-flight techniques. The ab initio structure determination of Bi_3ReO_8, assisted by information from several other physical methods, will be discussed. Examples in zeolite chemistry will include the use of Fourier techniques in the study of cation hydrolysis in lanthanum zeolite-Y, and an investigation of dealumination in ZK-5. The future scope of powder methods will be considered in the light of imminent developments at ILL, Grenoble and the spallation neutron source at the Rutherford Laboratory.