

integrated intensities extracted from a powder pattern and random starting phases) with a specialized framework search specific to zeolite structures, which can be described as 3-dimensional 4-connected topologies. The capabilities of FOCUS have been tested with six examples of medium to high complexity (zeolite topologies DOH, LEV, RSN, AFR, LTA, EMT), and the method was then applied to three novel zeolite structures - the two zincosilicates VPI-9 and VPI-10, and the beryllosilicate B2 - and a promising model was obtained in all cases. The structure of VPI-9 has since been confirmed with a full Rietveld refinement, and the code VNI has been assigned to that topology. Refinements for VPI-10 and B2 are in progress.

Experience shows that the approach of using chemical and geometrical knowledge can compensate for some of the information that is lost as a result of the overlap problem. At the same time, there is an intrinsic disadvantage: any method based on assumptions of certain structural properties is also limited to materials which conform to these assumptions. Examples which show the consequences of relaxing the structural assumptions were investigated, and it was found that the computing time requirements of FOCUS grow very rapidly with the number of different possible connectivity types. Suggestions for further developments to overcome this problem are outlined.

MS02.05.04 POWDER STRUCTURES FROM LIMITED DATA SETS. Damodara M. Poojary, Abraham Clearfield, Department of Chemistry, Texas A & M University, College Station, TX 7783

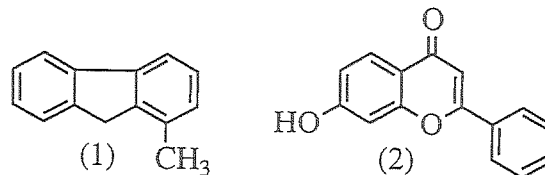
Structures of compounds which can be obtained only in the polycrystalline form depend mainly on the use of their powder diffraction data. Substantial progress has been made in instrumentation and computational aspects during the last decade for the application of powder diffraction techniques to solve unknown structures. One of the crucial steps involved in this process is to acquire the best possible diffraction data. Synchrotron sources, with their high brightness, excellent collimation and wavelength tunability provide optimum conditions for extracting individual intensities in the powder pattern. In most cases we have been able to arrive at the solution using data from a rotating X-ray source. However, synchrotron data was required to complete the structures in some cases.

This talk focuses mainly on the structure determination of metal phosphonates and phosphates. These compounds are difficult to obtain in single crystalline form and in most cases even their powder samples are poorly crystalline. The compounds yield only weak diffraction peaks whose intensities fall off very rapidly at higher scattering angles. Despite these difficulties, we have been able to solve the structures of a large number of compounds. Generally, the structures of these compounds are layered, where the metal-phosphate inorganic framework forms a two-dimensional layers which are separated by the organic groups on either side. In some cases metal phosphonate interactions led to unusual linear or porous structures. General methods used in solving the structures will be discussed using some representative examples.

In many cases the structures were solved by the use of 30-70 low angle (CuK α ; $2\theta < 60^\circ$) reflections by a combination of direct methods and heavy atom methods. The success comes from experience both in the structural aspects of these materials as well as the systematic application of efficient methods. It is equally important to use the results from other methods like spectroscopy, thermogravimetry, electron diffraction, etc. to arrive at the solution when only a limited number of powder diffraction data are available.

MS02.05.05 DETERMINATION OF MOLECULAR CRYSTAL STRUCTURES FROM X-RAY POWDER DIFFRACTION BY MONTE CARLO METHODS. MaryJane Tremayne, Department of Chemistry, UCL, London, WC1H 0AJ, U.K; Benson M. Kariuki, and Kenneth D. M. Harris, School of Chemistry, University of Birmingham, Birmingham, B15 2TT, U.K.

Many important crystalline solids cannot be prepared in the form of single crystals of sufficient size and quality for single-crystal X-ray diffraction studies, and in such cases it is essential that structural information can be extracted from powder diffraction data. We have developed and applied a method employing a Monte Carlo algorithm for crystal structure determination from powder diffraction data. In this method, a series of structural models is generated by random movement of a collection of atoms within the unit cell, and each trial structure is accepted or rejected on the basis of the agreement between the experimental powder diffraction pattern and the powder diffraction pattern calculated for the structural model. This technique differs considerably from the normal approach for structure determination from powder diffraction data, in that intensity information is not directly extracted from the diffraction pattern, and hence the problems of assigning intensities to overlapping reflections are avoided. The success of this method for *ab initio* crystal structure determination from X-ray powder diffraction data has been demonstrated by its application to the solution of several crystal structures including *p*-BrC₆H₄CH₂CO₂H [K. D. M. Harris, M. Tremayne, P. Lightfoot, P. O. Bruce, *J. Am. Chem. Soc.* (1994), 116, 3543] and the γ -phase of 3-ClC₆H₄CHCHCO₂H [B. M. Kariuki, D. M. S. Zin, M. Tremayne and K. D. M. Harris, *Chem. Mat.* (1996), in press]. In the work presented here, we have extended the Monte Carlo method to structure solution by a) simultaneous translation and rotation of a rigid structural fragment within the unit cell - illustrated by the structure determination of 1-methylfluorene (1) - and b) simultaneous translation and rotation with additional rotation about intramolecular bonds - illustrated by the structure determination of 7-hydroxyflavone (2).



MS02.05.06 CRYSTAL CHEMISTRY FROM POWDER DATA. Ian E. Grey CSIRO Division of Minerals, PO Box 124, Port Melbourne, Australia

A strength of structure analyses using powder data is the relatively short time required to obtain high quality data sets. This makes powder diffraction analysis particularly suitable for systematic structural studies on series of related compounds such as solid solutions in minerals and their synthetic equivalents. The high precision obtained when collecting and processing powder data sets under identical conditions allows the detailed study of subtle crystal chemistry correlations, for example variations in cation site occupancies, anion vacancies and bonding interactions.

The use of variable counting time (VCT) data collections (Madsen and Hill, 1994) enhances the capability to analyse subtle structure variations using X-ray data collected using a conventional laboratory configuration. In particular it yields more consistent thermal parameters, more accurate site occupancies and more stable refinement of light atoms.