

## 01-Instrumentation and Experimental Techniques (X-rays, Neutrons, Electrons)

developing viable initial models of solid state inorganic systems is outlined, with an emphasis on their application to topical materials problems. In addition to direct analogs of classical modeling-building methods, techniques for automated model-building, from polyhedra, cages or sheets, and for the automatic determination of unit cells and/or space group symmetries from such models are described. Simulated annealing, using simple geometric, potential, or diffraction-pattern matching functions is also proving effective as a direct space route to structure determinations from powder diffraction data. Recent results for aluminosilicate frameworks, mixed metal oxides, and molecular systems are described.

*Acknowledgement: The Biosym Catalysis and Sorption Project is supported by a consortium of industrial, academic and government institutions.*

**MS-01.05.02 ORIENTATIONAL ORDER-DISORDER TRANSITIONS IN THE VAN-DER-WAALS COMPLEX  $C_6H_6 \cdot C_6F_6$  - A CASE FOR COMBINING POWDER NEUTRON AND X-RAY DATA.** By Jeremy K. Cockcroft<sup>1</sup>\*, Andrew N. Fitch<sup>2</sup>, and Jeffrey H. Williams<sup>3</sup>, <sup>1</sup>Department of Crystallography, Birkbeck College, Malet Street, London WC1E 7HX, United Kingdom; <sup>2</sup>ESRF, BP220, F-38043 Grenoble Cedex, France; and <sup>3</sup>Institut Laue Langevin, BP156, F-38042 Grenoble Cedex, France.

Recent developments in powder diffraction during the last few years indicate that in future *ab initio* crystal structure solution will no longer be the sole domain of single crystal methods. In particular, the development of high-resolution powder diffractometers with sample environments routinely covering the temperature range 2 K to 1500 K at both X-ray synchrotron radiation and neutron reactor and spallation sources opens up a field of chemical crystallography of interest to both the synthetic and physical chemist. The developments in hardware are being mirrored by the production of user-friendly data treatment packages suitable for the non-specialist crystallographer. There is growing interest in the refinement of structures using more than one source of data, for example combining the information from neutron and X-ray data sets or combining X-ray data with molecular simulations.

This paper will discuss several systems showing the advances recently made in powder diffraction with respect to *ab initio* structure determination with emphasis on systems that needed both neutron and X-ray data for their solution and refinement. The phase transitions in the 1:1 complex formed by benzene and hexafluorobenzene will be used as one example of a system whose chemical crystallography can now easily be studied using high-quality powder data and modern software. The desirable attributes of modern Rietveld programs will be mentioned.

**MS-01.05.03 COMPLEMENTARITY OR COMPETITIVITY OF SYNCHROTRON AND NEUTRON POWDER DIFFRACTION.** A.W. Hewat, Diffraction Group, ILL, 156X Grenoble, Cedex, France.

The European Synchrotron Radiation Facility (ESRF) is becoming operational in Grenoble France, on the same site as the European High Flux Reactor (ILL). In the USA too, a new Synchrotron source is being constructed at the Argonne National laboratory, not far from the existing pulsed neutron source. It is hoped that the combination of the best synchrotron and neutron scattering facilities on the same site will stimulate complementarity between the two techniques. Certainly in times of budget constraints it will produce a certain competition.

Crystallography using neutrons will be among the areas most challenged by the new synchrotron sources. With such high X-ray intensities it becomes in principle possible to work with 'single crystals' of inorganic materials the size of the crystalline grains of powders. Alternatively, the high synchrotron intensity can be traded for very high resolution powder diffraction, while for neutrons, resolution remains limited in many cases by the limited intensity from even the best sources.

In this paper we will examine some of the recent successes of both neutron and synchrotron powder diffraction, and show that the two techniques are more complementary than competitive, and are likely to remain so in the immediate future. Neutron powder diffraction has evolved considerably over the years, and must continue to evolve to retain its place as an essential crystallographic tool for chemists. Synchrotron powder diffraction must also evolve to establish a rôle for itself, distinct from both conventional X-ray powder diffraction and neutron powder diffraction.

**MS-01.05.04 A MULTI-PURPOSE VACUUM DIFFRACTOMETER FOR OPERATION AT THE PHOTON FACTORY.** By S.W. Wilkins,<sup>\*</sup> Z. Barnea,<sup>†</sup> D.C. Creagh,<sup>@</sup> T.J. Davis,<sup>\*</sup> R. Garrett,<sup>#</sup> S. Janky<sup>†</sup> and A.W. Stevenson<sup>\*</sup>

<sup>\*</sup> CSIRO, Division of Materials Science and Technology, Clayton, Vic 3168, Australia. <sup>†</sup> School of Physics, University of Melbourne, Parkville, Vic 3052, Australia. <sup>@</sup> Physics Department, University College, University of NSW Campbell, ACT 2600, Australia. <sup>#</sup> ANSTO, Lucas Heights Laboratories, New Illawarra Rd., Lucas Heights, NSW 2234 Australia.

A versatile high-resolution two-axis diffractometer has been constructed for operation at the Photon Factory as an Australian National Facility [Barnea, Z. et al (1989) *Rev. Sci. Instrum.* **60**, 2537-40]. The instrument is capable of operation in various modes including:

- i) high-resolution powder diffraction with single-counter and crystal analyzer (Fig.1a),
- ii) high-resolution powder diffraction in Debye-Scherrer mode with imaging plates as recording medium, either stationary or translating for time-dependent studies (Fig.1c),
- iii) small-angle x-ray scattering (Fig.1b),
- iv) protein crystallography in screenless Weissenberg mode (Fig.1c),
- v) two-axis single crystal diffractometry using mono-detector or imaging plates (Fig.1a or 1c).

Some important features of the instrument are the capability for operation with all main components in vacuum and also the use of a double-crystal sagittal focusing monochromator as primary monochromator together with the optional use of a condensing-collimating channel-cut (CCCC) monochromator or other channel-cut as secondary monochromator. The use of a CCCC monochromator enables fine tuning of beam position on the sample, harmonic suppression, beam condensation and variation of wavelength band-pass [Wilkins, S.W. & Stevenson, A.W. (1988) *Nucl. Instrum. & Meths.* **A269**, 321-8. Wilkins, S.W. (1986) *Aust. and Int. Pat. PC1/AU87/00262*.] Further features include the use of high-precision absolute encoders on both shafts and the use of a large camera radius (570 mm) for the imaging plate cassette.

## 01-Instrumentation and Experimental Techniques (X-rays, Neutrons, Electrons)

25

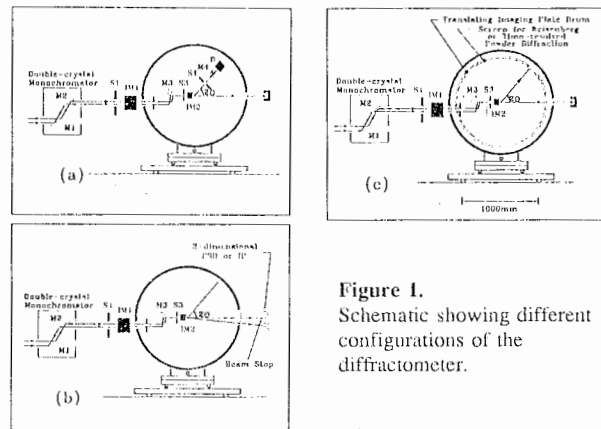


Figure 1. Schematic showing different configurations of the diffractometer.

PS-01.05.05 CATION DISTRIBUTION IN ZN-OXIDE SPINELS. By W. Schäfer<sup>1</sup>, G. Will<sup>2</sup> and J. Gal<sup>2</sup>, <sup>1</sup>Mineralogical Institute, University Bonn, Neutron Diffraction Group KFA Jülich, Bonn, Poppelsdorfer Schloß, 5300 Bonn, Germany; <sup>2</sup>Ben-Gurion University, Beer Sheva, Israel.

The cation distribution in 4 compounds:  $ZnAl_2O_4$ ,  $ZnFe_2O_4$ ,  $TiZn_2O_4$  and  $SnZn_2O_4$  has been determined by neutron diffraction. Part of the investigations were done by conventional monochromatic powder diffraction at the FRJ-2 reactor in Jülich, part by TOF-measurements at the spallation sources ISIS. In both cases a position sensitive linear detector has been used.

The diffraction measurements were preceded by Mössbauer studies, which yielded contradicting results. The question arose whether the compounds crystallize as normal spinels or as inverse spinels. Only neutron diffraction with sufficient differences in the scattering lengths between Zn and the other cations will give the proper result.

The analysis was done by the Two-Step-Method with first a profile analysis and profile fitting yielding intensities for each reflection. The actual analysis and the cation distribution was then done with the POWLS least squares/program. Procedures and results will be shown.

01.06 -- Open Commission Meeting on Neutron Diffraction  
-- Complementarity of Neutron Sources

## OCM-01.06.01

SCIENCE FROM PULSED SPALLATION NEUTRON SOURCES. By J L Finney, ISIS Facility, Rutherford Appleton Laboratory, Chilton, Didcot, Oxon, OX11 0QX, UK.

Conventional wisdom has it that pulsed neutron sources should be used when high energy and high momentum transfer is needed. This advantage over continuous reactor sources arises from the presence in the under-moderated spectrum of a significant high energy, low wavelength component, and facilitates high energy excitation studies and access to high scattering vectors. The time structure of the neutron pulse, however, allows a wide range of science to be performed that would be difficult, inefficient, or impossible on continuous sources. These advantages include very high resolution in both space and time, measurements over wide dynamic ranges of both energy and momentum transfer, the exploitation of fixed scattering geometries, and very low backgrounds.

These advantages have been exploited to great effect in areas of interest to crystallographers. Examples include the structure solution and refinement from powder data of systems for which single crystals were previously required, the detailed exploration of diffuse scattering, and the simultaneous measurement of both structure and dynamics in changing systems. Again contrary to conventional wisdom, pulsed sources are powerful sources of cold neutrons, and examples will be given of structural work exploiting long wavelength neutrons.

Although pulsed sources are relatively young, they have developed rapidly and are now established as powerful sources which have particular strong advantages. They are also capable of further development, as witnessed by proposals in both Europe and the USA for new sources up to 30 times the power of the ISIS source in the UK.

OCM-01.06.02 PULSED NEUTRON SOURCES AND STEADY REACTOR SOURCES

By Y. Endoh, Department of Physics, Tohoku University Aramaki Aza Aoba, Aoba ku, Sendai, 980, Japan

We have argued for a long time the necessity of the complementary usage of both steady and pulsed neutrons for condensed matter science in particular. In early days, we have developed pulsed neutron scattering techniques by carrying out experiments at the electron lineac facility of Tohoku University. Pulsed neutrons has been delivered since the late 60' s. In 1981, the first pulsed spallation neutron beam was delivered at the National Laboratory for High Energy Physics (KEK) in Japan after three year' s construction of this neutron facility, KENS. It was once the most intense and also the first full scaled pulsed neutron facility in the world. Since then KENS has been improved by the renewal of the spallation target as well as intensifying the proton accelerator, but it is a far small scaled facility compared with the world biggest neutron facility of ISIS at the Rutherford Appleton Laboratory.