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 system are described.

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MS–01.05.02 ORIENTATIONAL ORDER-DISORDER TRANSITIONS IN THE VAN-DER-WAALS COMPLEX C_6H_6:C_6F_6 – A CASE FOR COMBINING POWDER NEUTRON AND X-RAY DATA. By Jeremy K. Cockcroft[1]*, Andrew N. Fitch[2], and Jeffrey H. Williams[3], [1]Department of Crystallography, Birkbeck College, Malet Street, London WC1E 7HX, United Kingdom; [2]ESRF, BP220, F-38043 Grenoble Cedex, France; and [3]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 SYCHROTRON AND NEUTRON POWDER DIFFRACTION.
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The European Sychrotron 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,[+] Z. Barnea,[!] D.C. Creagh,[@] T.J. Davis,[+] R. Garrett,[#] S. Janky[+] and A.W. Stevenson[+]**
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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. PCT/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.

