

Society of Crystallographers in Australia



SCA

Newsletters

No 27, January 1995

The SCA homepage is located at <http://www.sca.asn.au>

FROM THE PRESIDENT

There has been a steady inflow of responses to Crystal XIX over the last few weeks, with about 80 registrations received. Particularly encouraging is the number of students who have applied for assistance to attend and who will give oral presentations or posters. The abstracts received cover diverse topics and the scientific programme is shaping up to be interesting and stimulating. Although the official date for registrations has closed, we will continue to welcome late responses for the next couple of weeks. A third circular, providing a map and campus plan, and a provisional programme, will be sent to registrants later in February.

A positive move in encouraging inter-country crystallographic collaboration was the decision by the Crystallographic Society of Japan to include a joint Australia-Japan symposium in their annual meeting last November. A number of Australian crystallographers attended and presented results of experiments carried out on the Australian National Beamline Facility (ANBF) at Tsukuba, and at the neutron facility at ANSTO. Hiroo Hashizume, who organised the symposium, has included an account in this *Newsletter*.

Hugo Rietveld was recently awarded the prestigious Gregori Aminoff Prize by the Royal Swedish Academy of Sciences in recognition of his development of profile refinement methods for the analysis of powder diffraction data. SCA members may be interested in his Australian connection. He gained a BSc(Hons) in Physics from the University of Western Australia and went on to do a PhD there with Ted Maslen as his supervisor. He carried out some of the first structure determinations in Australia using neutron diffraction data collected at Lucas Heights. This involvement contributed to his development of the Rietveld method, for which he was awarded the prize.

Of particular interest in this *Newsletter* is a report from the Australian National Committee for Crystallography prepared by John White. The report summarises major Australian contributions to equipment design at both the ANBF and at ISIS, the world's most powerful pulsed neutron source. The report also describes progress made by an ANCCr Working Party (now a Steering Group) in obtaining access to a third generation synchrotron source. There appear to be good prospects for access to beamlines at the Advanced Photon Source at Argonne National Laboratory, with financial support from the United States and from a number of Australian Universities. Over the last few months a number of Australian crystallographers in the Steering Group have put in an enormous effort to develop a detailed proposal to present to the government's new Major National Research Facilities scheme. It is this tireless behind-the-scenes work that leads to major benefits for the whole crystallographic community and their efforts are greatly appreciated.

As part of the ongoing series of sketches of crystallographic laboratories, Chris Howard has submitted an article for this *Newsletter* on the neutron facilities at ANSTO. Contributions have been rather thin on the ground recently. Sketches of other laboratories are welcomed for the coming newsletters, so that a complete picture of crystallographic research in Australia and New Zealand can be built up.

After a long and productive involvement on the Council of the SCA, Max Taylor has with reluctance tendered his resignation as Secretary, to concentrate on re-establishing after his recent operations. Mark Spackman has taken over the position, with Max kindly offering to maintain responsibility for the membership files. All Council members join with me in

thanking Max for his guidance and unstinting contributions to the SCA over many years. We look forward to his company at Crystal XIX.

A bientot, at Ballarat.

Ian Grey

FUTURE MEETINGS

CRYSTAL XIX. April 18-21, 1995 and THE BEVAN FEST, April 21-22, 1995 at The University of Ballarat (Mt. Helen campus), Victoria. A third circular will be available soon. For further information contact Drs Ian Grey or Lachlan Cranswick, CSIRO Division of Mineral Products, PO Box 124, Port Melbourne, Victoria 3207. Ph: 61-3-647-0211, e-mail: XTAL-19@dmp.csiro.au, fax: 61-3-646 3223.

AsCA'95, the second conference of the Asian Crystallographic Association will be held in Bangkok from November 22-24, 1995. For further information, and a request for a second circular, contact Professor Ward Robinson, Department of Chemistry, University of Canterbury, Private Bag 4800 Christchurch, New Zealand. E-mail: w.robinson@chem.canterbury.ac.nz, fax: 64-3-364-2110.

SKETCHES OF CRYSTALLOGRAPHY LABORATORIES

Neutron Scattering Group

Australian Nuclear Science and Technology Organisation

The Neutron Scattering Group is one of a number of groups at the Australian Nuclear Science and Technology Organisation's Lucas Heights Research Laboratories which is carrying out crystallographic research, or research heavily dependent on crystallography and crystallographic techniques. The Neutron Scattering Group (Dr Chris Howard), making use of neutron beams from the HIFAR research reactor, is in fact within the same program (Applications of Nuclear Physics) as the team led by Dr Richard Garrett, responsible for operating the Australian National Beamline Facility at the Photon Factory, Tsukuba, Japan. Crystallographic studies are undertaken within the Advanced Materials, Biomedicine and Health, and Environmental Science programs - these will be reviewed at a later date.

The specific role of the Neutron Scattering Group is to carry out scientific research making use of the thermal neutron beams from the HIFAR reactor. Most of this research involves collaboration with other institutions, especially Australian universities.

Single crystal studies are conducted under the charge of Dr Lindsay Davis. There are two four-circle diffractometers for single crystal work, 2TanA and 2TanB. The 2TanA diffractometer has the higher resolution, and this diffractometer has also been completely rebuilt during 1994. The new 2TanA is a Huber four-circle instrument, controlled by a PC-486 with locally developed Windows based software, and equipped with a cryorefrigerator (to 10 K). A furnace, for studies to 1000°C, is to be ordered shortly. Computer programs XTAL3.2, SHELXS and SHELX92 are available for data reduction and analysis. The instrument is used for classical neutron crystallography, such as the location of hydrogen atoms in complex crystal structures. A recent example, of work completed on the old 2TanA, is the elucidation of the role of hydrogen bonding in the formation of complexes between benzoic acids and cyclam (collaboration with Professor Len Lindoy and his team at JCUNQ).

There is a strong program in powder diffraction. The instrumentation at present comprises the high resolution powder diffractometer (HRPD) which is the responsibility of Dr Brett Hunter, and the medium resolution powder diffractometer (MRPD) being run by Dr Shane Kennedy. The HRPD is used for the study of more complex crystal structures, or high precision crystallography, whereas the MRPD tends to be used for phase transitions, magnetic structures, and time-resolved crystallographic investigations. Samples can be cooled (to 4 K), heated (to 1700°C), or held under pressure during the measurements. As regards the applications of powder diffraction, special interests of the group include high critical temperature superconducting oxides and related materials (Brett Hunter and Dr Michael James, in collaboration with staff at the Argonne National Laboratory and Cambridge University respectively), fast ion conductors (Lindsay Davis, working with Ron Balsys at the University of Central Queensland, and New Zealand scientists), zirconia and zirconia based ceramics (Chris Howard and Dr Margaret Elcombe, working with Drs Ray Withers and John Thompson at the Australian National University, Drs Steve Hull and Ron Smith at the Rutherford Appleton Laboratory, Dr Richard Hannink and his co-workers

at the CSIRO Division of Materials Science and Technology, Dr Erich Kisi at the University of Newcastle, and ICI Advanced Ceramics), metal hydrides (Margaret Elcombe and Shane Kennedy, with Drs Evan Gray and Erdong Wu at Griffith University, Dr Stewart Campbell at the Australian Defence Forces Academy, Dr Brendan Kennedy at the University of Sydney, and Erich Kisi), and magnetic structures and phase transitions (Lindsay Davis with staff from CSIRO Applied Physics; Shane Kennedy, with teams from the Monash University magnetism group led by Dr Trevor Hicks, Dr Peter Goodman and co-workers at the University of Melbourne, Dr Tim St Pierre at Murdoch University, and Stewart Campbell). The applications of neutron powder diffraction are supported by considerable in-house expertise in various methods for data analysis, including powder pattern indexing (Visser program), structure determination, and structure refinement by the Rietveld method (GSAS and LHPM). This expertise includes some familiarity with the construction of electron/nuclear density maps from X-ray/neutron powder data by the maximum entropy method. Complementary studies by X-ray powder diffraction (exploiting the access to the Photon Factory) and neutron powder diffraction are another current interest.

The neutron scattering group maintains further instrumentation, serving those with interests in what are perhaps more specialised areas of crystallography. This includes the triple axis spectrometer (Margaret Elcombe) for studies of excitations in crystalline solids, the long wavelength polarised neutron polarisation analysis instrument, Longpol, (Shane Kennedy) which is used for basic studies in magnetism and more recently for the investigation of magnetic flux pinning in superconductors and measurements on magnetic ribbons, and the small angle neutron scattering instrument, AUSANS, (Dr Robert Knott), now being commissioned. In addition, members of the group (Margaret Elcombe, Michael James) are assisting with the commissioning and operation of the new SURF neutron reflectometer at the ISIS facility, Rutherford Appleton Laboratory, U.K.

Members of academic institutions which subscribe to the Australian Institute of Nuclear Science and Engineering (AINSE) are reminded that they are eligible to apply for instrument time on the neutron scattering facilities. Enquiries should be addressed to Dr Roger Gammon, the AINSE Executive Officer (rgb@atom.ansto.gov.au) - the closing date for applications is September 30th. Access for collaborative research on topics of mutual interest is another option - enquiries about such access should be made directly to ANSTO Neutron Scattering Staff.

REPORT FROM THE NATIONAL COMMITTEE FOR CRYSTALLOGRAPHY

February 1995

Neutron Scattering Developments 1993-1995

The Australian National Committee for Crystallography surveyed Australia's needs for access to "Big Science" facilities in 1989 (*A Requirement for Australian Research Access to "Big Science" Facilities*, March 1989). The position taken by the ANCCr was supported in the 1991 ASTEC Report *Small Country, Big Science*. One of the positive outcomes was to give Australian researchers more formal access to ISIS, the world's most powerful pulsed neutron source. Financial support was provided by ANSTO and by the ISTAC travel grant scheme of the Department of Industry Science and Technology.

In 1994 the SURF project, to build the world's most intense neutron reflectometer at ISIS, has led to close collaboration between ANSTO and ISIS with a strong input to the design from the Research School of Chemistry, ANU, and the Department of Chemistry, University of Queensland. The SURF instrument will allow structure measurement on magnetic, surfactant and polymer monolayer and multilayer films at a resolution of a few Angstroms.

The Photon Factory, Japan.

The Australian National Beamline Facility (ANBF) at the Photon Factory is now almost fully operational. As a powder diffractometer it has unsurpassed speed and very high precision, good data sets being collected in twenty minutes on one recent campaign of experiments. The line is now working well for EXAFS and another workshop for EXAFS users is foreseen for mid 1995. A focussing monochromator using Australian technology was installed in December 1994. This has increased the intensity of the beam by a factor of 20 to rival the best at Tsukuba, thus making the instrumental performance required for protein crystallography attainable. The instrument has been oversubscribed at the two latest meetings of the Program and Review Committee. Further modifications to the focussing optics will be done in 1995, and half of the instrument time handed back to the Photon Factory as agreed in the Memorandum Of Understanding.

Plans for Access to a 'Third Generation' Synchrotron Radiation Source.

By early 1994 it had become obvious that Australian requirements for access to the Australian National Beamline at the Photon Factory would exceed the available beamtime once the Japanese host organisation started to use its agreed 50%

share of the resource.

Accordingly, the ANCCr established a Working Party which reported to the Academy of Science in November 1994. The Working Party's report *A Strategy for Australian Access to Synchrotron Beam Radiation* proposed that further developments be undertaken at the Photon Factory in parallel with a new initiative to gain access to a 'third generation' synchrotron radiation source. In the latter category, the sources recommended for detailed consideration were

- the European Synchrotron Radiation Facility (ESRF) Grenoble, France,
- the Advanced Photon Source (APS) Argonne National Laboratory, Chicago, USA, and,
- SPring 8, Japan.

The opportunities at ESRF and APS were immediate, attractive and quite different in style. Those at SPring 8 were of longer term.

Even before the ANCCr Working Party had completed its review, the Australian government announced a program of Major National Research Facilities designed to underpin Australian infrastructure in science and technology. The ANCCr Working Party was accordingly reconstituted as a Steering Group to develop a detailed proposal under the Australian government's new scheme. The Council of the Academy gave its general endorsement to the ANCCr strategy, and asked to see the final version of the proposal. However, ANCCr is aware that it would not be appropriate for the Academy to support any particular proposal.

The members of the Steering group were: Professor J.W. White (Chair), Dr J. Boldeman, Dr P. Colman, Associate Professor D.C. Creagh, Professor H.C. Freeman, Dr R.F. Garrett, Dr J.M. Guss, Professor F. Larkins, Dr R. Leckey, Associate Professor E.N. Maslen, Dr S. Wilkins.

In January 1995 the Steering Group made the difficult decision that the Advanced Photon Source (APS) is in principle, to be preferred as the site of an Australian 'third generation' synchrotron radiation facility. The mechanism for gaining access to APS is likely to be membership of three CARS consortia. Two of the consortia are led by the University of Chicago, and one by the Argonne National Laboratory. In reaching its decision, the Steering Group was particularly impressed by the prospect of gaining immediate, assured and Australian-controlled access to beamlines of the highest quality for protein crystallography.

The cost of the proposal will be \$8.7M over five years, with a contribution of approximately \$1.5M pa from the United States consortia. If the proposal is funded, Australia should have access to some of the world's best facilities for protein crystallography, for materials science, and for geoscience characterisation. Australian researchers should also have excellent opportunities to collaborate in the development of a new generation of focussing X-ray instruments for applications such as X-ray microscopy at variable wavelengths which are potentially of great value for biological and materials-science imaging.

The spectroscopic and X-ray optics communities were included from the outset in the deliberations of the ANCCr Working Party and in the development of the proposal. Interesting possibilities for research and student supervision in both areas were identified and are being followed up.

Following approaches from John White in his capacity as Chairman of the Steering Group, formal indications of support for the proposal were obtained from the Australian National University, the Universities of Sydney, New South Wales, Melbourne, Queensland and Western Australia, and Monash University. Through their respective Deputy Vice-Chancellors, the universities agreed to provide an immediate working capital of approximately \$200K pa over five years, and to make substantial commitments with respect to staffing, design and construction. This encouraging result bodes well for the proposal.

John White

AUSTRALIA-JAPAN SYMPOSIUM ON POWDER DIFFRACTION

The first Australia-Japan symposium on powder diffraction, *Impact of Powder Diffraction on Advanced Materials Research*, held during the Crystallographic Society of Japan 1994 annual meeting at Osaka University (24-26 Nov 94) was a great success. More than sixty participants attended, and participated actively in discussions, demonstrating the interest of Japanese crystallographers in the Australian National Beamline Facility at the Photon Factory. After a short introductory talk by Professor Hashizume, six presentations were presented at the symposium:

- The Multi-purpose Diffractometer on the Australian National Beamline at the Photon Factory. Dudley Creagh (Univ. of New South Wales);
- Initial Experiments with BIGDIFF on Beamline 20B at the Photon Factory and Prospects For Use in Powder Diffraction Application Studies. Brian O'Connor, A. van Riessen, G. Burton, J. Carter (Curtin Univ.), Richard Garrett and D. Cookson (ANBF, Photon Factory);
- Analysis of Powder Diffraction Collected on the Australian Beamline at the Photon Factory. D. Cookson, G. Foran, Richard Garrett (ANBF, Photon Factory), Brendan Kennedy (Univ. of Sydney) and Terry Sabine (ANSTO);
- Current Powder Diffraction Research Using Synchrotron Radiation in Japan. H. Toraya (Nagoya Inst. Tech.);
- Neutron Powder Diffraction in Australian Materials Research. Chris Howard, Brett Hunter and Shane Kennedy (ANSTO);
- Application of TOF Neutron Powder Diffraction to High-Tc Super-conductors. F. Izumi (Nat. Inst. for Res. in Inorg. Matter).

D. Cookson, Dudley Creagh and Shane Kennedy impressed the audience who believe that the high resolution, clean signal and speed of BIGDIFF, is not obtainable on any other beamline instrument. Dudley announced that the powder diffractometer using imaging plates would be considered to be operational in 1995 and open for general proposals. Chris Howard explained how neutrons diffraction can be used to complement X-ray diffraction in his masterful talk. Dudley Creagh gave a speech, which was well received, at the conference party. He described the past collaborations which enabled the creation and operation of the ANBF at the Photon Factory. The Australian presence in the Japanese crystallographic community is now well recognized. What needs to be pursued in future is the human interaction and a strengthening of scientific collaborations.

Hiroo Hashizume

CRYSTAL FRAGMENTS

Alison Edwards has taken up a lectureship in the School of Chemistry, University of Melbourne in October 1994. Alison completed a Ph.D. at the University of Melbourne under the supervision of Dr Bernard Hoskins in 1990 and has spent the last five years as a post-doctoral fellow at the Chemical Crystallography Laboratory in Oxford working with the Oxford Centre for Molecular Science and also with Dr S. G. Davies.

SUBSCRIPTIONS

The Treasurer wishes to remind members that subscriptions for 1995 are now due. A statement of the amount payable was distributed with the October 1994 *Newsletter*. Note that there is a discount if payment is made by April 1, 1995.

Graham Smith
