

## Society of Crystallographers in Australia



# SCA

### Newsletters

## No 31, May 1996

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# Sketches of Crystallography Laboratories

## New Zealand

### 1. The New Zealand Institute for Industrial Research & Development (IRL), Lower Hutt, New Zealand.

Industrial Research Limited (IRL) is one of the new government science companies that grew out of the Department of Scientific and Industrial Research (DSIR) divisions and the science research groups in the Ministries of Agriculture and Health. Chemistry Division, DSIR, traced its history back to 1865 (the Colonial Laboratory) before this restructuring in 1992.

X-ray diffraction equipment (Metropolitan Vickers) was first installed and operated by George Paterson in 1947. Early work on New Zealand clays was carried out with Morice Fieldes, later of Soil Bureau, DSIR. In 1957, the X-ray facilities were upgraded with a Philips powder diffractometer; Peter Williams began his studies on barbiturate identification and in 1959 went to Cambridge to complete a PhD in crystallography. Peter later lead the Physical Chemistry section which hosted a collection of spectroscopic techniques. Powder diffraction services provided "fingerprinting" for the forensic scientists and supported the mineral, geochemistry, and cement and concrete research groups e.g. developing quantitative methods for estimation of mineral phases in cements and rocks. Single crystal studies focused on studies of polymorphism, in support of the drug identification, and thermally induced phase changes in organic compounds. Others associated with this section were Prof Ray Golding, Dr David Natusch and Prof David Rae.

In 1969, thanks in part to some effective lobbying by Dave Rae prior to his departure to Australia, a Hilger & Watts Y290 was installed and lovingly maintained until the late 1970s by Dr Kevin Brown. Dr Graeme Gainsford joined DSIR in 1974 and immediately became a major user. As well as continuing to provide analytical facilities for the DSIR scientists (Carbohydrate & Organic Sections), collaborative small molecule studies were undertaken with Australian National (Professor Sargeson), Victoria (Professors Ferrier, Curtis and Halton), and Massey (Drs Brodie and Ainscough) Universities and data were collected by Dr Ted Baker (Massey). At the end of the 1980's, as hardware/electronic faults became regular, the decision was taken to concentrate on powder diffraction and to combine with the University of Canterbury in their 1982 purchase of a single crystal Nicolet P3 diffractometer for future data collections. In 1993-94, IRL confirmed its intention to maintain this excellent cooperation by contributing to the upgrade of the diffractometer to the Siemens P4 standard. The laboratory has one generator (Philips PW 1140) set aside with single crystal Precession & Weissenberg cameras for preliminary examinations.

The laboratory has continued to develop its expertise in X-ray powder diffraction supporting the Ceramics, Catalysis and Inorganic Materials, and Superconducting teams' research programmes. The Ceramics team lead by Dr Ian Brown operates the facility, still resident in D Block at the Gracefield Site. There are two diffractometers with automatic sample changes: a Bragg Brentano Philips PW 1050 with 32 sample changer run with Philips automation and processing plus local software on a Micro Vax II, including the JCPDS database. The second diffractometer has a 16 sample changer. The main generator is

also set up with a Huber Hagg-Guinier film camera, while the second generator has an Aton Parr HTK10 High Temperature furnace permanently mounted and automated with Sietronics Siexram microprocessor and software. Mr Martin Ryan is the scientist responsible for the running of the equipment, with computer support from Drs Mark Bowden and Graeme Gainsford.

## 2. University of Auckland

Chemistry Department, Auckland, New Zealand.

X-ray crystallography began in New Zealand in 1948, when F J Llewellyn (later Sir John) took up the chair of chemistry at what was then Auckland University College. Llewellyn's principal research interest was in organic nitrogen compounds, stemming from work on explosives during World War II. The foundation research student was Larry Calvert, (subsequently at National Research Council, Ottawa). His first task was to resurrect an abandoned dental X-ray unit and adapt it to single crystal work. The total other equipment of the laboratory comprised two Unicam single crystal rotation cameras. In 1949 the group was joined by Hugh Maslen and David Hall (both of whom remained at

*continued on next page*

Auckland), and the equipment was gloriously augmented by a Solus-Schall dual unit generator and a Unicam Weissenberg camera. Computing equipment comprised a Robertson strip-and-mask set, (a variation on Beevers-Lipson strips) and an analogue machine devised by Llewellyn for evaluation of sine-cosine products. Llewellyn remained until 1956, and his students included June Sutor (University College, London), Graeme Claridge (Soil Bureau, Wellington), Bryan Craven (University of Pittsburgh), Peter Williams (DSIR Wellington), and Neil and Joyce Waters (University of Auckland and Massey University). The group was subsequently led by David Hall, then by Neil Waters, and more latterly by Cliff Rickard and George Clark.

In 1969, a Hilger & Watts Y290 diffractometer with PDP8 computer was installed. This was replaced in 1981 by a Nonius CAD4, and was itself replaced by a Siemens SMART system in April 1996. The X-ray laboratory is housed on the fourth floor of the Chemistry Department, with current staff Drs C Rickard, G Clark and L-J Baker as technician.

The group has mainly specialised in structural studies of inorganic and coordination compounds but also has an interest in organic natural products. Recently G Clark has undertaken a number of studies of oligonucleotides which will be an ongoing project.

## 3. Massey University

Chemistry & Biochemistry Dept, Palmerston North, New Zealand

Following post-doctoral fellowships with Prof. Dorothy Hodgkin, Sylvia Rumball (1967) and Ted Baker (1970) joined the staff at Massey University to initiate a program in protein structure determination. In 1974 Ted Baker determined the structure of actinidin, the first protein structure to be determined in the Southern Hemisphere. In 1977, Sylvia and Ted published the first crystallographic characterisation of lactoferrin. Bryan Anderson joined the staff in 1977. It was not until 1982 that an independent X-ray crystallography laboratory was set up with the purchase of an Enraf-Nonius CAD4 diffractometer, for both small molecule structures (Joyce Waters) and protein structures (Ted Baker). In 1986, analysis of electron density maps was revolutionised by the arrival of an Evans and Sutherland graphics work station.

The next major event was the arrival in 1992 of the Rigaku RAxis IIC image-plate system and Rigaku RU200 rotating anode X-ray generator, which revolutionised X-ray data collection for protein molecules. Computing and graphics facilities now include 7 Silicon Graphics work stations. In 1996, low temperature capability was added to the macromolecular-structure facility. Gill Norris, who had been a research officer since 1983, joined the academic staff in 1993 and in 1994 Geoff Jameson, a former student of Ward Robinson's, joined the Structural Biology Group, after 12 years at Georgetown University. Over the years, Heather Baker has been a key figure in the art of protein crystallisation in the Structural Biology Group. Early in 1996, this group comprised 29 people, including graduate students, post-doctoral fellows, research officers and technician, and numerous sabbatical visitors. Tony Burrell, who joined the Department of Chemistry in 1994, has assumed responsibility for the small-molecule operations, on the departure of Joyce Waters in 1995. Geoff Jameson retains an interest in pathological (especially twinned) small-molecule structures.

## 4. University of Canterbury

Chemistry Department, Christchurch, New Zealand.

Professor Bruce Penfold was encouraged by Professor Cuth Wilkin's lectures to travel to Cambridge for his doctoral studies

in X-ray diffraction. He returned in 1952 setting up the laboratory through a generator grant in 1953 and a Weissenberg Camera built by Dick Nokes to an A.McL (Sandy) Mathieson design. First students were Stan Simpson (later head of WRONZ), Don Wright and Max Taylor. The first Weissenberg diffractometer (H. Freeman's lab design) was implemented by Graeme Gainsford in 1967 and supplanted by an Hilger & Watts Y290 with DEC PDP8 computer in 1969. In 1982, a Nicolet/Siemens P3 diffractometer was installed and became the first "totally in one room" system in New Zealand. It was shortly after added to with locally-modified Nicolet low temperature facilities. The system was upgraded in 1993 to the full Siemens P4 standard, with PC control.

Currently, about 300, usually low temperature, data sets are collected annually through Professor Robinson's close cooperation particularly with scientists at Universities of Otago, Waikato, Victoria (Wellington), Industrial Research Limited and also other research institutions. The X-ray laboratory is housed on the eighth floor of the Chemistry and Physics Building at the Ilam site.

## 5. University of Otago

Chemistry Department, Dunedin, New Zealand.

The mineralogical and petrological research of Prof D S Coombs and Dr W S Fyfe during the 1950's was given much needed analytical support with the purchase, jointly by the Chemistry and Geology Departments of a Philips X-ray diffractometer in 1957. This instrument supported research conducted by A J Ellis, R M Carr, A M Taylor, J Rogers, C J Churchman and others in a variety of areas ranging from zeolite synthesis, aluminosilicate stabilities and solubilities to coal chemistry and clay mineral reactivity. A new diffractometer system with a Philips PW1010 generator was purchased by the Chemistry Department in 1971 and the generator was replaced with a PW1830 unit in 1992. Since 1957 the powder equipment has been used mainly for "fingerprint" identification and for teaching basic X-ray crystallography. Currently R M Carr is using the equipment to examine the formation of aluminium oxide pillars in Vermiculite.

Single crystal work began in the early 70's with a collaboration between Jim Simpson and Brian Robinson from Otago with Bruce Penfold and Ward Robinson in Christchurch. The appointment of Claire Couldwell to a temporary post on the staff in 1974 brought and passed on first hand structure solving expertise to the Department, as well as a great deal of software. The Otago/Canterbury collaboration has stood the test of time with Otago making regular use of the excellent X-ray data collection service provided by Ward Robinson. The number of staff solving and refining their own structures has also grown, with Lyall Hanton, Sally Brooker and Allan Blackman all members of the burgeoning X-ray club.

## 6. University of Waikato

Chemistry Department, Hamilton, New Zealand.

When the School of Science at the University of Waikato was set up in 1970, a basic Philips powder diffractometer was purchased for teaching, and for some Earth Sciences and Chemistry research, it was up-dated to a Philips PW1840 in the mid - 80's. With the development of the material science group at Waikato there was a need for more sophisticated equipment, and in 1996 the Centre for Technology installed an automated Philips XPert powder diffraction facility complete with high temperature accessories. Operated under the supervision of Nigel Sammes, it is expected to renew interest in powder XRD research from others in the School.

Single-crystal X-ray studies at Waikato took the first steps in 1979, under the patient and benevolent eye of Ward Robinson, when it became clear that our synthetic inorganic and organometallic chemistry research projects needed access to structure determinations. Brian Nicholson became the coordinator and structure analyses are carried out routinely, with intensity data collection being done at Canterbury (Ward Robinson), or at Auckland (Cliff Rickard & George Clark). The local facilities consist of two precession cameras which are used for preliminary studies of crystal quality and for space group assignment.

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### THE AUSTRALIA PRIZE

The 1996 Australia Prize was awarded for work on *Pharmaceutical Drug Design* to SCA member and former President, Peter Colman, of the CSIRO Division of Biomolecular Engineering, Melbourne. Peter shared the prize with three other scientists; Dr Paul Janssen, Dr Graeme Laver, and Professor Mark von Itzstein. In part the citation to the award reads: "Dr Colman's use of X-ray crystallography to determine the structure of *sialidase* has been widely hailed as an intellectual *tour de force*. Now considered one of the world's leading X-ray crystallographers, Dr Colman conceived of inhibiting *sialidase*, a key protein on the surface of the influenza virus, to slow the replication of the virus. He was also a driving force in the

project which resulted in the design of the anti-influenza drug."

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## AsCA'95: students' views

(The following are reports from recipients of SCA travel grants which allowed them to attend the Asian Crystallographic Association (AsCA'95) meeting held from November 22-24 1995 in Bangkok).

### 1

Bangkok is a big bustling city, bursting with people. An action packed place, it is full of sights, sounds and smells. There are many reasons why people go to Thailand - there were two good reasons why I went: (a) AsCA'95 and (b) trekking around the hills north of Chiang Mai.

At AsCA'95 it was good to catch up with old friends, and meet new people. There were many interesting talks to attend - especially those about charge density and synchrotron radiation. While generally the most interesting talks tended to be related to one's own field, it was worth-while attending talks about other aspects of crystallography. Talks on new instrumentation, drug design and protein crystallography were interesting, although it was hard to grasp the finer details.

Due to a combination of fortuitous events, it was possible for me to attend the computing school. While not having that much to do with solving structures, it was interesting to learn the theory behind it. As the computing school was somewhat smaller than the main conference, it was easier to chat casually with some of the legends that were involved at the school.

No conference is complete without food - and while the food was generally great, the pineapple was superb. It had to be eaten to be believed. A bit of shopping and sightseeing was squeezed in around the busy conference schedule. A trip to the Grand Palace was an absolute must. Dripping in gold and jewels, it was an awesome sight. Numerous trips to the markets were also essential to stock up on those all important souvenirs (especially the T-shirts).

And the trek, it was fantastic - plodding through the jungle on an elephant, cruising down a river on a bamboo raft, puffing up and down hills - through the bush to the villages - a stark contrast to the concrete jungle (crowded with people) that is Bangkok.

*Barbara Etschmann*

### 2

It was with several cans of insect repellent, my quota of anti-malarial tablets and plenty of enthusiasm that I and twenty six other Australians (can a Cockney really call himself Australian?) embarked on AsCA'95 in November last year. Held at Chulalongkorn University in Bangkok from 22nd to 24th November, the conference brought together crystallographers from twenty countries across the Asian region. We were treated to a geography lesson early on in the extremely spacious and very well air-conditioned lecture room A (yes, it was a good idea bringing a jumper) on possible locations for future AsCA meetings by the IUCr President, Professor Philip Coppens. It appears that the venue has to be within several hundred kilometres of the centre of gravity of the Asian region so it remains a possibility that we could all be in Darwin in 2001.

The meeting proper opened up with a fine plenary lecture from Professor Ando from the Photon Factory on the applications of synchrotron radiation to crystallography and particularly interesting for me, a glimpse of what we may be doing in the future. This set the standard for the rest of the conference. With fifteen

microsymposia, it was not always easy deciding which lecture to attend, especially with the inevitable clashing of related subjects. Personally, I was glad to see that neutron diffraction and surface crystallography had not been scheduled for the same session.

There was a great number of very fine lectures and it was sometimes quite frustrating to have the speakers limited to fifteen minutes when many clearly could have gone on (and sometimes did). Well, I suppose that's what the coffee breaks are for. Playing devil's advocate, the chairmen I saw did a fine job keeping the whole conference running to time, which is never an easy task.

As with all conferences, some of the most interesting and useful information is learned outside the lecture room and AsCA was certainly no exception. It was also most interesting comparing PhD experiences with counterparts from other countries in the region and certainly many new friends (and potential future collaborators) were made.

And then there are the poster sessions with over 150 presentations in three days. I was scheduled for the first day and was quite hoping for a number of probing questions to be asked relating to my work. Something along the line of 'have you tried this technique?' or 'have you tried analysing your data this way?' In retrospect I think the most intriguing I received was 'how do you produce your colour print outs?'

The Farewell Banquet was a perfect mixture of food, drink and conversation. There seemed to be some bias towards one of these categories on the mostly ANU table which, with the help of a couple of fellow PhDers from the University of Melbourne, did a fine job of relieving the catering staff of all the Singha beer within a 500 metre radius of the venue. This was followed with a very successful attempt by Professor Phathana Phavanantha of the local organising committee to find a representative of each of the twenty countries to say a few words. After all the Singha put away on my table, it was most fortuitous that Chris Howard spoke on behalf of the Australian representatives.

Of course, there was also Bangkok at night. A range of visits were made to some of the local drinking establishments and also the Hard Rock Café where a few of the delegates really excelled themselves with some pretty stylish dancing.

To close, Professor Phavanantha, all the local organising committee, Professor Ward Robinson, Dr. Jim Simpson and soon to be Dr. Jan Wikaira, along with the international organising committee (a list far too long to mention individually) really do need to be congratulated for all the many hours of hard work which were involved in making the conference the great success it was.

Very finally, I would like offer my sincere thanks to the SCA Council for providing me with a generous travel grant to attend what was, as you can no doubt tell from the above, a very interesting, stimulating and extremely enjoyable conference.

*Elliot Gilbert*

### 3

Things did not bode well for our conference aspirations as we boarded a taxi in Khao San Road and asked to be taken to the venue of the AsCA'95, Chulalongkorn University. Our accents were so bad that the Taxi driver thought we meant Chiang Mai, the northern capital of Thailand. Fortunately we managed to avoid the long ride north but it was a pleasant surprise when we eventually got to the university that the student accommodation of Vitaya Nives Hall was more like a luxury apartment complex than a shack -which had been our most recent experience of Bangkok hotels.

Although Damian was laid up with a gastric virus on the first night, the welcoming reception was lovely - an oasis of food and drink on a typically balmy Bangkok evening. The traditional Thai dancing was fantastic but the food took the cake, so to speak. In fact, it seemed that every AsCA'95 social function, formal and informal, was adorned with that magnificent Thai cuisine. However we had not come to Bangkok just for the food, and the broad range of presentations were enough to whet the most jaded intellectual appetite. Of that range we can mention only a few. The plenary lecture given by Takao Matsuzaki was excellent. The method that he described of designing molecules that could interact with the active site of a protein was ingenious.

It was particularly exciting for both of us to hear Gautam Desiraju's lecture, whose work on the utilisation of strong and weak hydrogen bonds to design molecular networks was of relevance to our fields of study. He showed how subtle changes to a molecular structure can dramatically affect the three-dimensional architecture. The lecture also encompassed an excellent exposé of the information it is possible to extract from the Cambridge Structural Database.

It was hard to single out an Australian presentation, as we had a very strong contingent, but the lecture given by Siegbert Schmidt, who elegantly described the general methods and his own results in determining modulated structure, deserves a mention. Of the posters, we found Graham Smith's particularly interesting, being another example of the use of hydrogen bonds to direct the three-dimensional structure.

Presentations aside, it was a great opportunity to meet and socialise with fellow crystallographers, not only from Australia, but from all over Asia. The socialising culminated in the final dinner in which we found solace for the lack of beer with the cheery company of the ANU representatives.

Alison and Damian would like to express their gratitude to the SCA for providing the opportunity to take part in AsCA'95. In addition, Alison is particularly grateful for the chance to attend the Asian School on Crystallographic Computing. At this School several very generous and patient delegates delivered lectures and tutorials on fundamental aspects of crystallography.

*Alison Green and Damian Slizys*

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## FASTS

FASTS (Federation of Australian Scientific and Technological Societies) Council is a lobby group comprising numerous Scientific and Technological Societies (including RACI, AIP, ANZSCB, SCA, etc) organised into 9 sections (Biological, Earth, Mathematical, Chemical, Marine, Medical, Physical, Plants & Ecology, Associate members) which lobbies politicians, business etc. on behalf of ~40,000 working scientists across Australia. It produces regular newsletters, circulars, press releases, reports and speeches. These and other up to date information on its constitution, policies, office bearers and its member societies can be obtained on the World Wide Web site <http://bimbo.pharmacol.su.oz.au/fasts/fasthome.html>.

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## FUTURE MEETINGS

### CRYSTAL XX

This is the first announcement of the Twentieth Meeting of the Society of Crystallographers in Australia which will be held from April 2-5, 1997 Queenstown, Central Otago, New Zealand. The first circular for this meeting is in preparation and will be available on the World Wide Web in the next fortnight. All members of the SCA will be advised, by e-mail, when the page is posted and both e-mail and printed versions will be available from the organisers. To fit in with the Australian University Calendar the above dates have been arrived at. As they fall inside the University of Canterbury's academic year (where the organisers are based) a venue outside the university has been negotiated. The venue will be the Lakeland Hotel, Queenstown. Queenstown, as many of you will already know, is one of the most scenic and interesting parts of New Zealand. Nestled at the foot of the Remarkable Range on Lake Wakatipu it is not only a place of incredible beauty but the heart of adventure tourism in New Zealand. This includes being the home of bungy jumping.

## IUCr XVII Congress

August 8-17, 1996, Seattle, Washington, USA

The Seventeenth International Congress of Crystallography will be held in Seattle, USA, from 8-17 August 1996. Early registrations closed on June 1 but late registrations will be accepted. Up to date information can be obtained from the World Wide Web at <http://www.bmsc.washington.edu/iucr/>.

Included is information on registration, travel awards, accommodation, the main and affiliated meetings and on Seattle City. The Conference Abstracts are also listed.

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## ASIAN SCHOOL ON CRYSTALLOGRAPHIC COMPUTING

25-28 November 1995

Chulalongkorn University, Bangkok, Thailand

The School was organized jointly by the IUCr Computing and Teaching Commissions as a satellite of AsCA'95 (2nd Meeting of the Asian Crystallographic Association) in Bangkok, where the Local Organizing Committee was chaired by Prof. P. Phavanantha.

The School program included three half-hour lectures in the mornings, 45 minute lectures (mostly dedicated to computational aspects) and two-hour practical sessions in the afternoons. The morning lectures covered all basic aspects of crystallographic computing (data treatment, Patterson and Fourier, direct methods, refinement and interpretation of results) with also some introductions to more advanced topics such as extensions to macromolecular crystallography, treatment of powder data, analysis of thermal motion and charge density studies. The afternoon lectures gave an introduction to operating systems, and programming languages, an overview of crystallographic files for data exchange and publication submission, some ideas on the use of crystallographic databases, and some basic concepts on computer graphics and networks. The practical sessions were mainly hands-on usage of a rather wide variety of crystallographic software running on PC's.

Handouts with lecture notes were distributed to the participants and discussions and questions were encouraged. The lecturers who contributed to the School were: P. Coppens, G.R. Desiraju, Fan Hai-fu, C.M. Gramaccioli, S. Hall, C. Kennard, P. Phavanantha, M. Ramanadham, W.T. Robinson, J. Simpson, B. Skelton, H. Toraya, D. Viterbo and T. Yamane. The organizational aspects were coordinated by Ahpisit Ungkitchanukit.

The number of participants was 42 including 3 from Australia; their qualifications ranged from postgraduate students to assistant professor, but most participants were at the postdoctorate level. An anonymous questionnaire was distributed and 17 participants answered. Most people found the School useful and the practical sessions were mostly appreciated. The main problem was the very different crystallographic background of the participants (some answers to the questionnaire suggested a prior streaming of participants).

The atmosphere of the School was warm with very friendly relations among participants and lecturers, despite the intense program and the hard work. The generous coffee breaks and lunches organized by our hosts and, most of all, the practical sessions helped to establish this friendly climate which stimulated people to work harder and discuss their problems. The practical sessions always lasted more than two hours and some people had to be kicked out when the computer rooms had to be closed!

Finally, the success of the School was made possible by:

- the financial support of IUCr and of the Crystallographic Society of Japan,
- the enthusiasm of the Bangkok LOC and in particular the dedication of Dr. Ungkitchanukit (a theoretical physicist who volunteered to help us),

- the invaluable help in the setting up and advertising of the School given by the AsCA president, Prof. W. Robinson, and by Prof. J. Simpson,
- the encouragement of the IUCr president, Prof. P. Coppens and of Professors Ohashi and Uragami, responsible for AsCA'95,
- the work and dedication of all the lecturers who contributed to the School without any financial support, prepared the lecture notes and the practical sessions,
- the enthusiasm of the participants who indicated appreciation for our efforts,
- the warm hospitality of Chulalongkom University and the fascinating and friendliness of our Thai hosts.

*Prof. Davide Viterbo*

Chairman of the IUCr Commission  
on Crystallographic Computing

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## Society of Crystallographers in Australia

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