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# CRYSTALLOGRAPHY NEWS

## British Crystallographic Association

No.78

September 2001

BCA Spring Meeting 2002

Dorothy Hodgkin - RSC Landmark

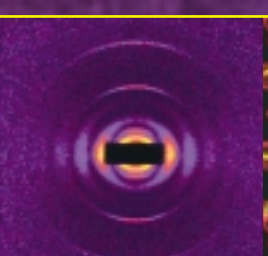
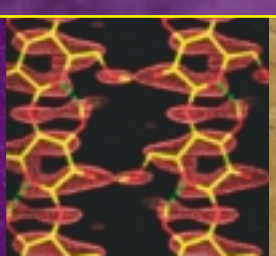
Fibre Diffraction

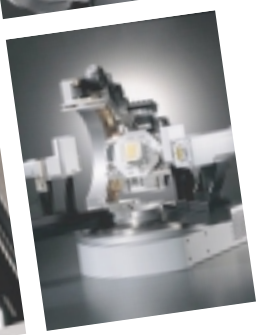
Crystallography and Antiquities

2001 Walter Hälg Prize

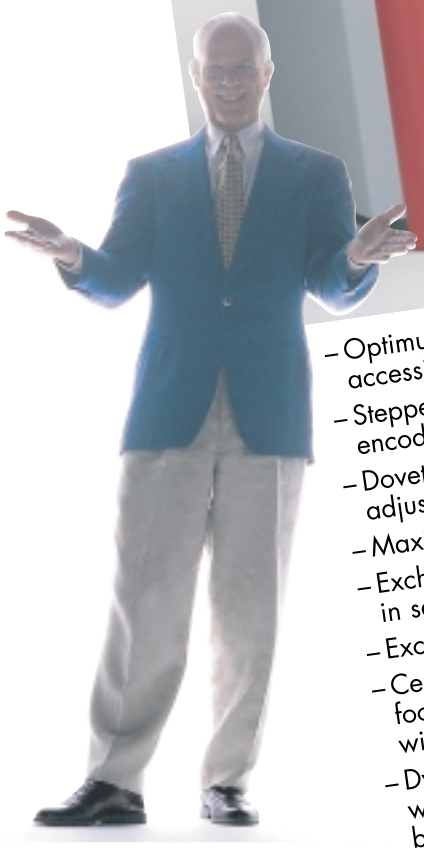
Book Reviews

Quarterly





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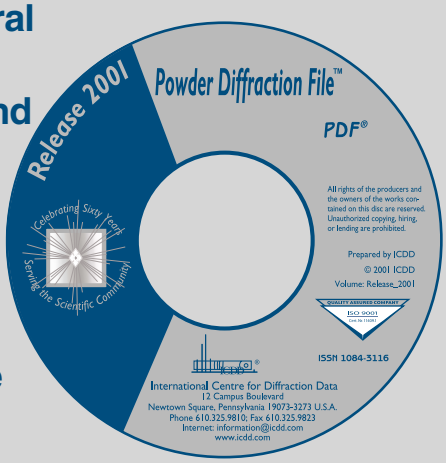


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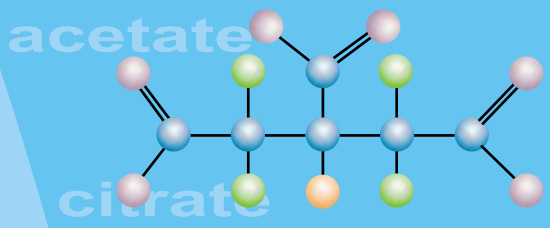


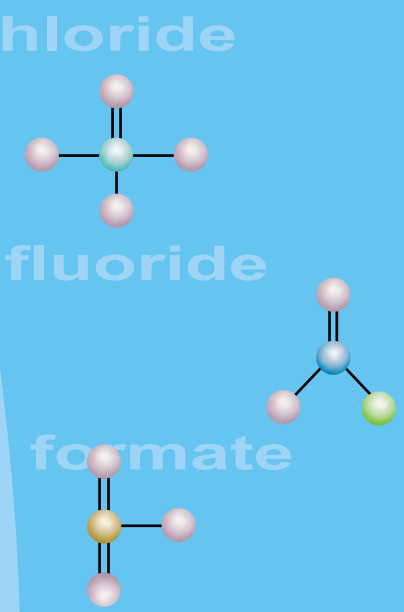


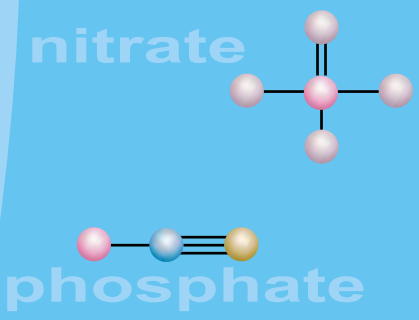


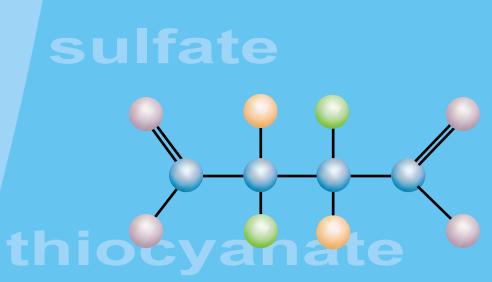


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## NEXT ISSUE OF CRYSTALLOGRAPHY NEWS

**CRYSTALLOGRAPHY NEWS** is published quarterly (March, June, September and December) by the British Crystallographic Association. MSword97 documents (or earlier versions) **may be sent on a PC disk or electronically**, (only small files < 500K please, image formats JPEG, GIF). Items may include technical articles, news about people (e.g. awards, honours, retirements etc.), reports on past meetings of interest to crystallographers, notices of future meetings, historical reminiscences, letters to the editor, book, hardware or software reviews. Please ensure that items for inclusion in the **December 2001** issue are sent to the Editor to arrive before **26th October 2001**.

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*The British Crystallographic Association is a Registered Charity (#284718)*

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# *President's Remarks*

Cover pictures left to right:

X-ray diffraction image of normal breast tissue.

2Fo-Fc map calculated from cellulose II.

Detail of Middle Kingdom coffin from El Bersheh.

Chinese underglaze-blue porcelain dish, mid-14th century, Yuan Dynasty.

Dorothy Hodgkin plaque.

*The conference season is upon us, and even in non-crystallographic meetings, crystallography is everywhere to be seen. I was at a Gordon Conference on 3-D Electron Microscopy in Rhode Island in June ( I was one of just three crystallographers!), and was amazed at the work in which low resolution particles whose shape was determined by single particle electron microscopy studies were filled with structures taken from the protein data bank thus giving a model of macromolecular assemblies at an atomic resolution. Studies of muscle contraction, for example, at this level are simply astonishing, and the microscopists have their sights set on whole cells and organelles at a similar resolution.*

*Other developments proceed apace: powder diffraction can now relatively routinely solve structures ten times the size of those 5 years ago, especially organic molecules even using laboratory data; the flood of biological macromolecule structures continues unabated (More than 14000 new structures were deposited in the protein data bank last year.); data collection hardware continues to improve and computing has become a minor cost. Crystallographic software becomes increasingly sophisticated and easier for non-experts to use (although this has obvious dangers). Diamond, the new synchrotron, is on the way, and plans are afoot for a new neutron spallation source; these will keep the UK in the forefront of crystallographic technology for the next decade. Crystallography has always been an exciting science, but it seems to me that the excitement is increasing and not in any way diminishing. Despite our many reservations about the way in which government and others handle and manage science, it is a great time to be a crystallographer.*

*Our own spring meeting has to reflect all this, and the organisers of our next meeting in Nottingham have done a splendid job: the plenary session is devoted to structure solution and phasing as applied to all systems from small molecules to macromolecular assemblies using both single crystals and powders. There are sessions on DNA recombination and repair, polymorphism, drugs and disease, Reitveld refinement, detectors, thin films, crystallisation, education and the formation of a new special interest group devoted to the Diamond facility. Many of these sessions will take the form of workshops. Every attempt has been made to keep costs to a minimum, and the Nottingham campus is a delightful place. How can anyone resist?*

*I hope to see many of you at the Krakov ECM Meeting at the end of August. ECM conferences also reflect the scope and diversity of 21st century crystallography as well as the BCA Spring Meetings.*

**Chris Gilmore**
**August 2001**

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Full committee details on the BCA website - **http://bca.cryst.bbk.ac.uk/BCA/**

## *The third issue*

*Have you ever wondered what goes on behind the scenes at the British Museum? In this issue Andrew Middleton describes how XRD plays a part in determining how, when and where antiquities were made. In contrast the article on Fibre Diffraction by Trevor Forsyth, ILL covers applications such as a diagnostic method for the investigation of cancer tumours in breast tissue and the study of hydrogen bonding in cellulose. Also in this issue there is a report on the Presentation of the Royal Society of Chemistry Landmark to Commemorate the Work of Dorothy Hodgkin.*

**Jo Jutson**
**April 2001**

## *BCA Spring Meeting 2002*

### The University of Nottingham, Monday 25th - Thursday 28th March 2002

The next BCA Spring Meeting will be held on the University Park campus of the University of Nottingham. An exciting scientific programme, characterised by a large number of joint sessions between the BCA groups, has been drawn up by their representatives. This includes a Plenary Session on new methods of structure solution and phasing, along with other sessions on DNA recombination and repair; polymorphism and structural changes; proteins, drugs and disease; new laboratory sources and detectors; thin films, and workshops on topics such as macromolecular crystallisation, Rietveld refinement, the CRYSTALS program … and much more. So whether you want to hear the latest results, find out about new techniques and instrumentation, hone your skills in the workshops, or simply catch up with old friends, we are sure you will be able to enjoy doing all of these.

The University Park campus has extensive, attractive green space and even boasts a boating lake. On-site cultural attractions include the Arts Centre and the brand new D.H. Lawrence Pavilion. Accommodation in halls of residence is available within a few minutes walk of the lecture theatres and exhibition halls, all of which are situated in the Pope Building. The social programme comprises a mixer reception on Monday evening, a sponsored wine reception/poster session on Tuesday evening and the conference dinner on Wednesday.

We think we have a great programme of scientific sessions, and a superb venue for the Spring Meeting in 2002. Full details of the Meeting will appear in the December issue of Crystallography News: the Meeting should be particularly attractive to students, who will qualify for free registration. In addition to the city itself, Nottingham is also an excellent base for visiting a wide range of attractions, from the scenery of the Peak District to historic cities like Lincoln. We look forward to welcoming you here next March.

**Sandy Blake**
(email: *A.J.Blake@Nottingham.ac.uk*)

**Claire Wilson**
(email: *Claire.Wilson@Nottingham.ac.uk*)
Local organisers

# *People in the News*

## Second European Crystallography Prize Awarded to Professor Jochen R. Schneider

The European Crystallographic Association has awarded the second European Crystallography Prize to Professor Jochen R. Schneider of the HASYLAB at DESY, Hamburg, Germany. Professor Schneider is recognised for his pioneering work on the application of gamma-ray spectroscopy and his high energy synchrotron radiation studies, as well as his more recent involvement in the development of the free electron laser.

Professor Schneider was born in Burgstädt, Saxony, Germany and studied Physics in Hamburg after an education as electrical engineer. He did his PhD under the direction of Professor H. Maier-Leibnitz at the Institute Laue-Langevin in Grenoble, France. His work on gamma-ray diffractometry and Compton scattering was performed at the Hahn-Meitner-Institut in Berlin, the synchrotron radiation work at DESY-HASYLAB in Hamburg, where he is now heavily involved in the development of free-electron lasers driven by linear accelerators. Professor Schneider is presently Head of HASYLAB and Director of Research for Synchrotron Radiation and Free-Electron Lasers at DESY.

## OBE for Professor Julia Higgins

Congratulations to Professor Julia Higgins OBE FRS, who became Dame Commander of the British Empire in the Queen's Birthday Honours list. Julia Higgins is Professor of Polymer Science in Imperial College. She also chairs the Athena project on women in science, engineering and technology (SET) in Higher Education in the UK, along with an impressive list of other responsibilities both to the SET community and for women in SET.

## Fellows of the Royal Society

Congratulations to Dr. Andrew Leslie and Professor George Sheldrick who have been appointed Fellows of the Royal Society.

**Dr Leslie** is Senior Scientist at the MRC Laboratory of Molecular Biology, Cambridge. He has determined the atomic detail of a number of biologically important structures and most recently solved the structure of hepatitis B virus protein.

**Professor Sheldrick** is Professor of Structural Chemsitry at the University of Gottingen and Director of the Institute fur Anorganische Chemie, Gottingen, Germany. He has been a major contributor to the field of chemical X-ray crystallography for the past three decades and developed the SHELX computer programs, for structure determination and refinement.

## New ICDD Executive Director

The International Centre for Diffraction Data (ICDD) have announced that Dr. Tim Fawcett has joined the ICDD as its new Executive Director. Tim is a long time ICDD member, ICDD Fellow, and served on the Board of Directors from 1988-1990. He brings to the ICDD outstanding experience in management and R&D for product development, as well as an exceptional background in X-ray diffraction.

## The J.D. Hanawalt Award

Mr. Raymond P. Goehner, and Dr. Joseph R. Michael, both of Sandia National Laboratories, Albuquerque, New Mexico, U.S.A. have been selected to receive the 2001 J.D. Hanawalt Award for excellence in the field of X-ray powder diffraction. The J.D. Hanawalt Award is presented every three years for important, recent contributions to the field of X-ray powder diffraction and phase identification published within the last five years. The award consists of a commemorative plaque, an honorarium, and travel funds to attend the meeting at which the award and lecture will be presented.

## Retirement - Ian Langford

Dr Ian Langford, Reader in Powder Diffraction, University of Birmingham, has retired after working for almost 40 years with

Powder Diffraction, 21 of them with Arthur (Prof. A.J.C.) Wilson - 5 in the Viriamu Jones Laboratory, Dept of Physics, Univiversity College, Cardiff and then in the Physics Dept (now School of Physics & Astronomy) at Birmingham. Ian is a leading international expert in high resolution X-ray powder diffraction applied to many materials of environmental, technological and industrial interest and is the author of many papers on Powder Diffraction. He was a founder member of the BCA and was involved at the outset in establishing the Industrial Group. Ian served on the IG Committee, as an academic representative, from 1983 to 1989 and was the Group's first Secretary/Treasurer (1983/87). He was also a member of the BCA Council from 1985 to 1992, as the Association's Treasurer from 1988 to 1992.

*Acknowledgements*
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**The British Crystallographic Association is grateful to Birkbeck College, University of London, who host and manage the server for our Website.**

# *Presentation of the Royal Society of Chemistry Landmark to Commemorate the Work of Dorothy Crowfoot Hodgkin*

On 14 May 2001 the Lecture Room of the University Natural History Museum, Oxford was full of people eagerly anticipating the presentation of the second UK Royal Society of Chemistry landmark. The proceedings were chaired by Professor Graham Richards, Chairman of the Department of Chemistry in the University of Oxford. The Vice-Chancellor of the University, the historian Dr.C.R.Lucas welcomed us to the dedication. He reminded us that Dorothy was the first British Woman to win a Nobel prize in Chemistry in 1964. The University takes pride in her achievements which have contributed to the success of the Chemistry Department in Oxford, the largest in the country. He was sure that Dorothy would have been proud of the fact that some 40% of today's students are women.

One of Dorothy's students, Professor Sir Tom Blundell, FRS, then spoke on "*Structural Biology and Crystallography today: the influence of Dorothy Hodgkin on current developments*". (Reported later in this article) In introducing the speaker, Graham Richards reminisced that he and Tom had been undergraduates together at Brasenose College, and now Tom was Dorothy's second most famous student. (the most famous being Margaret Thatcher (nee Roberts) who later abandoned chemistry for law and became Prime Minister).

Professor Tony Ledwith of the Royal Society of Chemistry then presented a memorial plaque to Professor Richards who received it on behalf of the University. Since Oxford weather is unpredictable, a virtual unveiling was projected on the screen. We saw crimson velvet curtains slowly open to reveal the plaque which was fixed to the wall on the archway of the main entrance to the Inorganic Chemistry Laboratory in South Parks Road. The inscription reads:

## National Historic Chemical Landmark

The work of Dorothy Crowfoot Hodgkin at the University of Oxford.
"*In this building from 1956-1994 and at other times in the Oxford Science Area, Professor Dorothy Crowfoot Hodgkin (1910-1994) OM, FRS, Nobel Laureate, led pioneering work on the structures of antibiotics, vitamins and proteins, including penicillin, Vitamin $B_{12}$ and insulin using X-ray diffraction techniques. Many methods for solving crystal structures were developed taking advantage of digital computers from the very earliest days. The work provided a basis for much of present day molecular structure driven molecular biology and medicinal chemistry.*"
**14 May 2001**

Outside the Museum with the plaque
Left to right Dr C R Lucas, Vice Chancellor, Oxford University, Professor Graham Richards, Chairman of Chemistry, Oxford, Professor Tony Ledworth, Immediate Past President, Royal Society of Chemistry, Professor Sir Tony Blundell

A drinks reception followed courtesy of GlaxoSmithKline.
Left to right - Dr David Giachardi, General Secretary, Royal Society of Chemistry, Elizabeth Hodgkin, Professor Keith Prout

## "Structural Biology and Crystallography today: the influence of Dorothy Hodgkin on current developments"

**Professor Sir Tom Blundell FRS**
(Sir William Dunn Professor of Biochemistry at the University of Cambridge)

Professor Blundell began by describing Dorothy Hodgkin's early work in Cambridge with her Ph.D supervisor, J.D.Bernal, determining the structure of protein crystals using X-rays. In 1934 she took high quality X-ray photographs of the crystalline protein pepsin, having realised that the crystals had to be kept in their mother liquor if they were to retain their order. The study of proteins in water has been immensely important over the years. The final paragraph of the paper in Nature which she and Bernal published demonstrates remarkable foresight.

*"At this stage such ideas are merely speculative, but now that a crystalline protein has been made to give X-Ray photographs, it is clear that we have the means of checking them by examining the structures of all crystalline proteins, arriving at far more detailed conclusions about protein structure than previous physical or chemical methods have been able to give."*

Pepsin proved to be a member of a large family of proteolytic enzymes called the aspartic proteinases which have been extensively studied since then. They have about 320 amino acids and 2 motifs of Asp-Thr-Cly at the active site. Charles Bunn was one of the first to work on rennin which was also found to be an aspartic proteinase containing this same motif; it is involved in the inhibition of angiotensin II synthesis, and may be a possible target for better drugs to reduce high blood pressure. Pepsin has an extended deep active site and drug companies began to think about discovering drugs to fit it. In the 1980s chemistry dominated drug design, X-ray crystallography was traditionally used to model the 3D structures. Knowledge of the structure allows the design of structure based inhibitors but more recently it has proved cheaper to generate anti-hypertensive drugs using genomics.

The pepsin motif, Asp-Thr-Cly, was also identified in 1985 in the retroviral genomes of RSV and HIV, published by Toh H et al. in *Nature* **315,** 691 (1985). Studies of the life cycle of HIV and the structure of HIV proteinase led to the production of the drug 'Indinavar', an HIV proteinase inhibitor. The 3D structure of the HIV proteinases was discovered in 1989 but later mutations were found to have occurred, so cocktails of inhibitors had to be used to improve the treatment of AIDS. Pre-clinical Drug design now has a new paradigm since the publication of Complete Genomes in the public databases in December 2000. (Details can be found on the Internet at http://igweb.integratedgenomics.com/GOLD/)

Dorothy's interest in people led her to work on the structure of insulin, she felt she had to find the structure of insulin before a cure could be found to save children dying from early onset diabetes. This structure was published by Adams, M.J., Blundell, T.L, Dodson, G.G, Dodson,E, Vijau, M, and Hodgkin D.C et al. in *Nature* **224**, 491-495 (1969). Just knowing the structure did not satisfy Dorothy, she wanted to know how the insulin carried out its function in the body. Fred Sanger showed her a model of the insulin receptor binding which she admired but she had to understand how the insulin behaved in the body. She asked 'How does it work?' No one could answer that question then and despite more recent work by Hubbard and Hendrickson in 1995 and work by Louise Johnson on the structure of phosphorylase we still cannot answer Dorothy's simple question. Traditionally, laboratory X-rays were used to determine the structure of proteins and their 3D structures modelled. Today more powerful synchrotron radiation sources are mostly used. Structural biology is now being applied to aid pre-clinical drug discovery to find cures for diseases caused by stray mitogenic activity, and those involved in cell development, proliferation and differentiation. Modern computers and new algorithms using a drug like virtual library now speed up the process of screening new drugs more effectively. Dorothy would have enjoyed using these; she always tried to use the latest equipment in her research.

Perhaps Dorothy Hodgkin's most important influence was the way she worked and interacted with people. Although she collaborated with many who worked in industry, she never thought of this as collaborating with a faceless industry, but rather as working with her friends who happened to be employed by an industrial company. Her students learnt the importance of a large network of contacts to successful research.

**Kate Crennell**
**July 2001**

**Note:** Information about Dorothy Hodgkin can be found on the BCA web site, at http://bca.cryst.bbk.ac.uk/BCA/obits/CVS/DCH.html
and there is an excellent life history at:
http://www.engr.psu.edu/wep/EngCompSp98/Aclausi/HodgkinD.html

# *Fibre Diffraction: highlights for X-rays, neutrons and CCP13*

## Fibre Diffraction with X-rays and neutrons

As with many other fields, the scope of fibre diffraction is now changing very rapidly. Major developments in molecular biology/biochemistry mean that new biological systems are becoming available and that totally new approaches exist for sample preparation. There are parallels with this in material/polymer science. Exciting opportunities for the study of these systems are arising at X-ray and neutron beam sources. New facilities planned such as the DIAMOND synchrotron and the second target station at ISIS will be of major importance. Equally important will be the upgrade of existing facilities to exploit their full potential. At the Daresbury SRS, a purpose designed high-angle fibre diffraction camera has been constructed by Dr. R. Keyhoe for use on beamline 14.1 (see http:// www.dl.ac.uk/ SRS/ PX/ line14/ 14_1/fibre_camera.html). At the Institut Laue Langevin (ILL) in Grenoble a major refurbishment of instrumentation in under way as part of the *ILL Millennium Programme*: perhaps the most important development here for the fibre diffraction community is the planned upgrade of the D19 diffractometer – the new detector

on this instrument will give a gain in detecting solid angle of approximately 25 and open up entirely new possibilities for fibre diffraction (as well as single crystal) work (see http:// www.ill.fr/ YellowBook/ D19/ help/ dev/ development.html).

Fibre diffraction has made critical contributions to our understanding of biological and synthetic polymer systems in the past. The following highlights from recent X-ray and neutron fibre diffraction work illustrate the importance of the technique for the future.

## X-ray fibre diffraction – new insight to breast tumour tissue from the Daresbury SRS

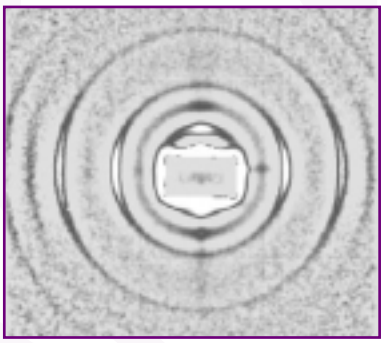

Figure 1. (a) Apparently normal breast tissue

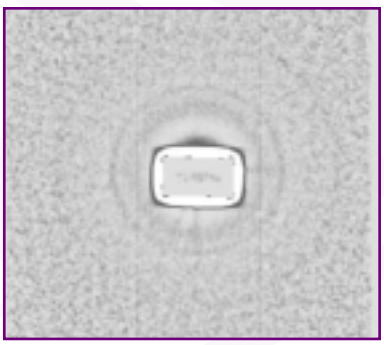

Figure 1. (b) Tumour tissue. X-ray diffraction images of 2 samples from the same breast. Most of the features present arise from collagen in the sample (see Lewis et al, J. Synch. Rad. 7, 348).

Recent results from beamline 2.1 at the Daresbury SRS suggest that X-ray fibre diffraction can be used as a diagnostic method for the investigation of cancer tumours in breast tissue. The study, which was published by Lewis, Rogers, Hall, Towns-Andrews, Slawson, Evans, Pinder, Ellis, Boggis, Hufton & Dance (*J. Synch. Rad.* 7, 348), shows clear differences between fibre diffraction patterns recorded from healthy and diseased breast tissue (see Figure 1). These differences are believed to occur as a result of changes in the structural ordering of the collagen within breast carcinomas. Collagen is a major component of the extracelluar matrix (ECM) in breast tissue; its degradation is known to be of major importance in the morbidity and mortality of cancer. Furthermore, extensive alteration of the ECM has been observed in other forms of cancer – collagen derangement has been attributed to enzymatic degradation and altered neosynthesis. Since the fibre-forming collagen molecules produce well-defined diffraction data, changes in the structural ordering of the ECM can be studied by X-ray diffraction. This approach offers some advantages over standard histopathology in that it utilises untreated samples and takes an average over all tissues illuminated by the X-ray beam. It is therefore potentially much faster and also less prone to errors that can occur by simply looking at the wrong part of a sample.

## Neutron fibre diffraction at the Institut Laue Langevin - hydrogen bonding in cellulose

In the neutron arena, one of the most striking results in fibre diffraction is the work of Langan, Nishiyama & Chanzy (*J. Am. Chem. Soc.* 121 (43), 9940) who are using unique facilities available for neutron fibre diffraction on instrument D19 at the Institut Laue Langevin (ILL) in Grenoble to study hydrogen bonding in various forms of cellulose. Cellulose is often said to be one of the most abundant polymers on Earth. These workers are interested in the structural basis of the biological, chemical and physical properties of cellulose. Central to this is the hydrogen bonding network of the hydroxyl groups of the polymer. Neutron diffraction provides the most powerful method that can offer a detailed visualisation of this hydrogen bonding network in cellulose fibres. Figure 2 shows a 2Fo-Fc map derived from data recorded from cellulose II samples that have been deuterated by mercerisation in NaOD. The maps clearly show the labile hydrogen atoms involved in the hydrogen bonding and allowed the authors to distinguish between two competing models for cellulose II.

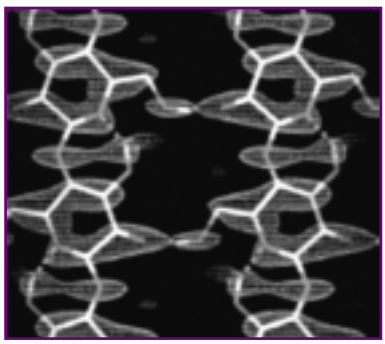

Figure 2. 2Fo-Fc map calculated from cellulose II. The neutron fibre diffraction data were recorded on instrument D19 at the Institut Laue Langevin, Grenoble (see Langan, Nishiyama, Chanzy, J. Am. Chem Soc. 121(43), 9940)

## The CCP13 collaborative computing project for fibre diffraction

The unique experimental opportunities available to the fibre diffraction community are of course being paralleled by seemingly relentless increases in computing power as well as major initiatives such as the "computational GRID" which aims, within a relatively short time, to revolutionise distributed computing and data transparency. These massively enhanced facilities for computing now mean that biological and synthetic polymer questions can be addressed in a way that was quite impossible even a few years ago. It is fortunate therefore that the fibre diffraction community has a well focused strategy for data extraction and analysis in the form of the CCP13 collaborative computing project. The project, which is now nearly 10 years old, has been exceptionally successful. Heavily allied to the NCD programme at Daresbury, it has grown from a community that was initially centred on X-ray fibre diffraction studies of biological molecules to one that now covers X-ray and neutron diffraction concerns over the whole polymer community. It is perfectly poised to deliver the radical new approaches needed for data extraction and modelling software to its biology and materials science communities.

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# *Crystallography and Antiquities*

In the last issue of *Crystallography News*, David Rendle (2001) introduced his article by saying that Crystallography in Forensic Science usually implies the use of X-ray powder methods (XRD). The same can be said really for *Crystallography and Antiquities*: rarely are single crystal methods used, though there have been exceptions, and the interest is most often in the qualitative identification of crystalline phases. Another parallel with the use of XRD in forensic science is, perhaps, the range of materials encountered: we might be analysing Anglo-Saxon jewellery one day and lumps of slag, sherds of pottery or Egyptian pigments the next. The parallels do not stop there either, for we too are interested in tracking materials back to their source, much as the forensic scientist might need to determine the source of a flake of paint or a trace of soil. However, there are differences and most of our XRD analyses are aimed at answering questions along the lines: *What was it made of?*, *How was it made?*, and *Where was it made?*, perhaps providing the information to address questions like *When was it made?* and *Is it a fake?*.

A common factor that runs through nearly all analyses carried out in the museum is that we are restricted in the size of sample that we use, so that for us XRD powder methods generally means the use of Debye-Scherrer powder cameras, rather than a diffractometer. Samples are normally removed under a binocular microscope offering maximum precision and minimum damage in sampling. A casualty of this type of sampling, of course, is any possibility of meaningful quantitative analysis. In a few case study 'cameos' I will try to provide some idea of the way XRD is used to help answer the questions just set out. Frequently, XRD provides only a part of the answer and usually the investigation will be collaborative, involving several different analytical techniques and perhaps several scientists.

Recently, we have been investigating the pigments used to decorate ancient Egyptian coffins and other artefacts (Middleton and Humphrey *in press*). Studies like this allow us to develop our knowledge of the ancient Egyptian artist's palette and the ways in which it changed over time and in different parts of ancient Egypt. XRD is well-suited to this type of investigation, allowing the identification of the pigment using only a minute sample, which can be removed almost non-destructively from an existing blemish on the 'paintwork'. Indeed, X-ray powder photography was used more than sixty years ago, by Jope and Huse (1940), to characterise a series of samples of Egyptian Blue, the blue pigment used on many artefacts from ancient Egypt (see, for example, the Middle Kingdom coffin shown in Figure 1). They were able to show that blue pigments from several sites in England were the same as examples from Egypt, and subsequent research has

confirmed that this pigment was used widely across the ancient world from about 3100 BC through the Roman period until it ceased to be used around the 9th century AD. Egyptian Blue is a copper calcium silicate, which is identical to the mineral, cuprorivaite. However, this is very rare in Nature and there is no doubt that the blue used in ancient Egypt and elsewhere was manufactured by heating together quartz, limestone or calcite and a source of copper (such as copper alloy filings) to a temperature of about 850-1000° C. It is the earliest synthetic pigment.

Figure 1. Detail of Middle Kingdom coffin from El Bersheh.

Natural minerals also formed an important element in the ancient Egyptian palette, including commonly occurring minerals such as hematite for red, limonite for yellows, and calcite or gypsum for white. However, XRD has revealed the use of some more unusual minerals. Jarosite, an iron sulphate, was used for a distinctive pale lemon-yellow paint on some Middle Kingdom coffins from Asyut in Middle Egypt, and there is increasing evidence that huntite, a magnesium calcium carbonate was used extensively as a fine, white pigment. Huntite was recognised as a mineral species only in 1953 (Faust 1953) and, because of its typically very fine grain size, it can be very difficult to identify either optically or by chemical analysis. However, the XRD powder pattern is diagnostic.

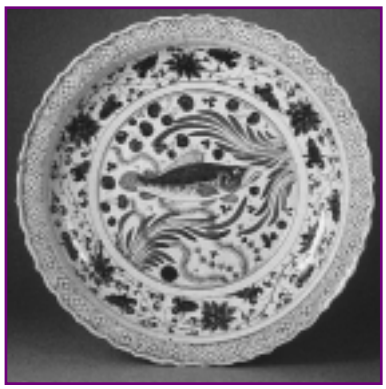

Figure 2. Chinese underglaze-blue porcelain dish, mid-14th century, Yuan Dynasty.

Chinese porcelain (Figure 2) became known in Europe from about the fifteenth century AD and almost immediately attempts were made to emulate it by European manufacturers. Chinese porcelain was made from a mixture of kaolinite and a 'porcelain stone' or 'petunze'. This provided the essential ingredient, a high proportion of sericite mica, which acted as a high temperature flux, vital to the successful manufacture of hard-paste porcelain. European manufacturers were unaware of this and used a variety of 'recipes', each with a different combination of raw materials. The mineralogy of these 'soft-paste' porcelains reflects the raw materials used by the various factories. X-ray powder photography, using minute samples removed from existing breaks or from an unglazed foot-ring (Bimson 1969), provided a means to characterise the nature of the paste mixtures used to make early English porcelain samples and compare them with Chinese porcelain. The so-called glassy-frit porcelains, which typically have calcium-rich compositions, were found to contain a calcium silicate mineral (typically wollastonite); those based on the use of magnesium-rich soapstone (talc) typically contain enstatite, formed by the transformation of the soapstone; and bone-ash porcelains were found to be characterised by whitlockite, a calcium phosphate mineral formed by the transformation of bone. By contrast, XRD showed that the Chinese hard paste porcelains are characterised by mullite, usually with some relict quartz and cristobalite.

Figure 3. Ninth century AD silver strap-end from Whitby with niello decoration.

Niello, a black material used to produce striking inlaid designs on decorative metalwork, has a long history as a jeweller's decorative technique, going back at least to the Romans. It has been identified in Anglo-Saxon jewellery (Figure 3) and its use extended across Europe and into

Asia but by the sixteenth century its popularity in Europe had waned. Nevertheless, some fine examples were made in Russia during the 18th and 19th centuries and the technique enjoyed a brief 20th century revival in England with the Arts and Crafts movement. Niello is made by melting metal filings with sulphur; various metals have been used in different places at different times, including silver, copper and lead, either singly or in combination. After cooling, the resulting metal sulphide is crushed, mixed with a flux, applied to the prepared area and then heated to soften (or melt) the sulphide into place, rather like an enamel.

Figure 4. Inlaid silver cup from Enkomi, Cyprus, c. 1400 BC.

Without some form of analysis, niello is easily confused with other black inlays, especially on ancient artefacts that may have been buried and corroded. It is here that XRD comes into its own, providing an unequivocal means by which the characteristic metal sulphides can be identified. For example, it has been possible to identify a mixture of silver, copper and lead sulphides on a fragment of shiny black inlay from the Enkomi cup (Figure 4), a fine example of Late Bronze Age, Cretan craftsmanship, dating to c. 1400 BC, suggesting, along with other evidence, that the antiquity of this technique may be considerably greater than previously thought (La Niece 1998).

Figure 5. Mesopotamian cylinder seals of serpentinite (larger seal on left, Akkadian, c. 2300 BC) and chlorite (right, Ur III period, c. 2100-2000 BC).

Cylinder seals (Figure 5) developed in Mesopotamia, alongside the cuneiform system of writing, beginning some time around 3500 BC. They have intricately carved designs on their curved surfaces and were usually perforated along their length, so that they could be suspended on a thread and worn. When rolled out in soft clay, the intaglio designs (carved in reverse!) provided a mark of the owner's authority, in much the same way as sealing wax and seals are still used on official documents. The cylinder seals were made from variety of materials, chiefly natural minerals and stones, but sometimes ivory, metals or faience. Identification of the materials of the seals can throw light on the exploitation of raw materials in antiquity but identification is not always as straightforward as anticipated and some form of analysis is essential to avoid misidentifications. The two seals shown in Figure 5 are very similar in appearance but analysis by XRD showed that they were made from two different materials (Sax et al. 1993). The larger, Akkadian period seal on the left (c. 2300 BC) was made from serpentinite, a stone well-suited to seal-making because it is fairly hard and takes a good polish. The seal on the right, which dates to the later Ur III period (c. 2100-2000 BC), was made from chlorite, a softer and much inferior material. Why change to an inferior material? The change coincides with an invasion of the area by a people called the Guti and it seems that this disrupted the trade routes that supplied the serpentinite, obliging the seal-makers to substitute it with chlorite, which was available more locally.

As noted at the beginning of this article, XRD may not always provide the complete answer to the questions we ask of antiquities but I hope that this short note has illustrated that it may often contribute something to our understanding of the ancient world.

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**Department of Scientific Research**
**The British Museum**

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# *The dehydration of α-lactose monohydrate*

## IG Poster Prize Winner, Reading 2001

α-Lactose monohydrate (ALM) is an extremely important pharmaceutical excipient. It is widely used because of its availability in a highly pure crystalline form, and because of its low toxicity. Lactose (β-D-galactopyranosyl(1,4)-C-glucopyranose) is the main sugar of milk and other dairy products. It appears as different forms, depending on the α- or β-glucose isomerization in aqueous solution, and the association of water molecules in the solid form. In aqueous solution, the two forms (α- and β-) are balanced by mutarotation. The natural crystalline state is α-lactose monohydrate, but several other solid forms exist, such as, anhydrous β, and stable and unstable anhydrous α-lactose. There is also an amorphous form of α-lactose monohydrate. The different forms have different hydration properties, and the physical transformations of lactose need to be characterised in order that, when used in a pharmaceutical formulation, it will remain stable. A change in form can lead to adverse changes in the active component of a formulation.

The object of this work was to investigate the dehydration of α-lactose monohydrate, using Molecular Dynamics modelling. Differential Scanning Calorimetry and X-ray Diffractometry were used during the course of the work, as characterisation tools.

## DSC and XRD results

The samples of ALM used in this study were dried in an oven under a stream of dry nitrogen gas, at various temperatures, before DSC and XRD traces were recorded. The DSC trace for a sample of lactose heated to 120°C shows very little change from that for unchanged ALM. A peak at 140°C is due to loss of water from the crystal lattice. At temperatures up to 160°C, DSC traces show no peak for the loss of water, but a peak around 170°C, suggesting a change to another phase. Drying ALM at 165°C results in a DSC trace with no peaks present, suggesting that this phase change takes place during the drying procedure at 165°C. The XRD results confirm this, with the pattern for a sample dried at 120°C being identical to standard ALM. According to the XRD trace for a sample heated at 150°C, the unit cell for this sample is similar to that for standard ALM, but the molecules are orientated differently.

## Comparison of Surfaces 020 and 100

The water in face 020 is arranged in layers between rows of lactose molecules, whilst the water in face 100 is arranged in channels perpendicular to the face. The way in which the water molecules are arranged affects the way in which they leave the structure of ALM when it is heated.

## Molecular Dynamics Simulations

The simulations were carried out at 420K, using the Hoover thermostat and Dreiding potential set throughout. NVT dynamics were done. The simulation of surface 020 suggested that the face goes through an amorphous face during the drying phase. This happens before the water molecules move away from the surface. As the topmost layer of this surface is composed of lactose molecules, with a layer of water beneath, this would seem to be necessary, in order for the water molecules to escape. The topmost layer was first to become disordered, followed in turn by lower layers. Face 100 also becomes disordered during the drying process, but to a lesser degree than face 020. This may be due to the channels of water present in this face, which would appear to allow the water molecules to leave this surface more readily than face 020. The molecules of lactose are more likely to stay in their original orientation in this face as well, whilst in face 020 they tend to move through 90°, so that the

two rings of the sugar molecule are arranged horizontally, rather than vertically.

## Conclusions, Discussion and Future Work

The results from DSC and XRD studies show that ALM dehydrates at 140°C, to give an unstable product. This is an unstable anhydrous form of α-lactose, which is very hygroscopic and converts readily to ALM. The peak seen on DSC traces for samples dried at temperatures lower than 165°C is caused by the conversion of this unstable anhydrous product to stable anhydrous α-lactose. At dehydration temperatures greater than 165°C, the product of dehydration is the stable anhydrous form, so there is no peak on the DSC trace corresponding to the conversion from unstable to stable anhydrous form.

The modelling results suggest that there may be an amorphous phase involved in the dehydration mechanism. This may be involved in the formation of the unstable anhydrous α-lactose. The samples used in this study were dried under nitrogen gas, in order that the same conditions could be maintained for all samples. Dehydration in a moist atmosphere may result in different products at different temperatures. To model the effect of water on dehydration, Molecular Dynamics simulations will be done with the surface of lactose in contact with water molecules. On-line dehydration, using a heating cell, will also be done, in order to investigate the kinetics underpinning the dehydration process.

H.L.Williams[1], G.B.Thomson[1], P.C.Wright[1], D.R.Merrifield[2]

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# *Book Reviews*

## The Basics of Crystallography and Diffraction - 2nd Edition

By Christopher Hammond

***Publisher*** Oxford University Press

May 2001
Hardback ISBN 0-19-850553-1, £49.95
Paperback ISBN 0-19-850552-3 £22.95

This text book is aimed at advanced undergraduates and post graduate students working in the areas of solid-state chemistry, physics, materials science and earth science, and also contains information that will be of value to more experienced research workers and lecturers. In this new edition the first edition has been significantly expanded (242 pp to 331) and much of the material has been revised and updated. In particular, the reader is introduced to topics that are of more general interest but that are closely related to the basic concepts of crystallography and diffraction.

By comparison to the first edition, in the second edition Chapter 1 is expanded to show how a variety of more complex crystal structures can be explained in terms of different faulting sequences of close-packed layers. In addition, the structures of carbon including fullerenes are discussed, and related to the development of symmetry ideas. In Chapter 2 the figures have been updated and these facilitate a clearer understanding of two-dimensional and, consequently, three-dimensional symmetry. Non-periodic patterns are also introduced. Chapter 3 now includes a short discussion on space-filling polyhedra and in Chapter 4 the discussion of space groups is significantly expanded, and is a very helpful background to the understanding of the Space Group representations in Vol. A of the *International Tables for Crystallography*. Chapters 5 and 6, covering lattice planes and Miller indices, and the reciprocal lattice, have been revised, and are now easier to read and understand. In Chapter 7 the human eye as an optical instrument is discussed, and the processes involved are related to the situation in the diffraction process for other wavelengths of electromagnetic radiation. In Chapter 8 the contributions of von Laue, the Braggs and Ewald to X-ray diffraction are described. Chapters 9 and 10 in the first edition have been expanded to Chapters 9, 10 and 11 in the new edition. The topics of X-ray and neutron diffraction from ordered crystals, preferred orientation and its measurement are now covered in detail, and the relevance of the work on preferred orientation to

materials and earth sciences emphasised. Chapter 12 contains an excellent discussion and explanation of stereographic projections, and the usefulness of the method explained. The book is completed with a series of useful appendices including details of crystallographic model builders, crystallographic software, biographical notes on famous crystallographers and other scientists working in the area of diffraction, useful crystallographic relationships, vectors and complex numbers, and systematic absences. There are useful teaching exercises at the end of each chapter with quite detailed answers at the end of the book.

Overall, the second edition represents a significant improvement on the already high quality first edition. The material is clearly laid out and the subject is developed logically. The fundamental importance of symmetry is made at an early stage. While the subject matter is correctly described as 'basic' the text can be read at several levels and there are aspects that will be of interest to undergraduate students and others that are better suited to researchers with greater experience and expertise. Throughout, the text is very readable, and the level of mathematics is appropriate to the subject matter covered. The additional material of preferred orientation fibrous materials is very interesting and useful, and is the discussion of electron diffraction and its applications given in Chapter 11.

I strongly recommend this book to students and lecturers working in the area of crystallography and diffraction. The textbook is one in a series sponsored by the International Union of Crystallography and published by Oxford University Press.

**Paul Raithby**
**University of Bath**
**July 2001**

## Nobel Prize Women in Science. Their Lives, Struggles and Momentous Discoveries

by Sharon Bertsch McGrayne

***Publisher*** Carol Publishing Group 1998 2nd Edition UK price £15

*ISBN 0-9702256-0-1* 450 pages, paperback with line diagrams and photographs

This is a fascinating book for all those interested in the history of science, especially those who wonder why some people become great scientists and yet others with apparently similar abilities leave science altogether. This book describes the lives of 15 women who have either themselves won a Nobel prize or played a crucial role in a Nobel-prize winning project; it tries to answer the question: why have only 10 of recipients of the Nobel Prize in the sciences been women when there have been over 300 men since the Prizes were first awarded in 1901?

The first chapter, '*A Passion for Discovery*' analyses the qualities these women had in common which enabled them to overcome the many obstacles in their way. *First, they adored science*, they had many other activities but they were all passionately determined and in love with their work.

*They had sympathetic parents and influential relatives;* most of them were from professional or academic families; for example we learn that Dorothy Hodgkin's uncle asked a friend for advice as to how Dorothy could continue in science after her first degree. The friend replied she should try to become a student of J.D.Bernal.

*Religious values stressing education were critical;* some were Quakers and half were Jewish.

*Behind many of these successful women stood a man; the* Braggs and J.D.Bernal encouraged a generation of English women crystallographers, including Dorothy Hodgkin. Others were not so fortunate, on page 7 we read '*Unfortunately for Jocelyn Bell Burnell's career her thesis adviser failed to become her mentor and she received little or no career counselling.*'

*Good luck and timing were vitally important;* pioneers like Marie Curie and Lise Meitner came of age just as European Universities opened their doors to women; eight of the fifteen women were born within fifteen years of each other, eleven within a single generation between 1896 and 1921. Some, like Gertrude Elion, (prize winner in 1988) had worked for years without managing to get a 'scientific' job, but it was not until late in the Second World War that the shortage of industrial chemists

enabled her to get a research post with Burroughs Wellcome. There she worked on purines, studying not only their structures but also their biochemical reactions, searching for better drugs for diseases such as leukaemia and others to prevent rejection in transplanted kidneys.
The book has 3 sections, 'First generation pioneers', including Marie Curie and Lise Meitner, 'Second generation' which includes both Dorothy Hodgkin and Rosalind Franklin, and 'The New Generation', Jocelyn Bell Burnell, an astronomer who discovered quasars as a graduate student, and Christiane Nüsslein-Volhard, a developmental biologist who gained the Nobel prize for physiology and medicine in 1995.

Each woman's life is described in a separate chapter using information from primary and secondary sources and interviews with colleagues, students, family, friends and experts in the field. Bibliographic references for the sources are collected into a section of 'Notes' at the end and there is a comprehensive index. All scientific explanations are nontechnical and illustrated with simple diagrams or photographs, including some I had not seen before for the crystallographers.

I enjoyed this book; I learned new facts on the careers of some people I thought I knew all about and as a physicist I was surprised to find I understood the more biological and medical chapters.

**Kate Crennell**
**July 2001**

# *Meeting Reports*

## Eighth Intensive Course in X-ray Structure Analysis

**30th March – 7th April 2001, Trevelyan College, Durham**

The Eighth Course was the third one to be held in Durham, the courses having begun in Aston University, Birmingham, in 1987. This course generally followed the successful format of its predecessors, but it has evolved to reflect changes which have occurred in structure analysis, a process which will continue. David Watkin, the director of the first five Courses, made a welcome return in the role of lecturer, replacing Bob Gould who had directed the previous two Courses in Durham.

The Course is based on lectures given by a group of four lecturers, interspersed with tutorials in small groups of eight students: this time we had nine groups, a total of 72 students. All lecturers and tutors are present for the duration of the Course, and so are available for informal questions or discussions: it is common to see tutors sacrifice at least part of their coffee breaks to help students with particular queries. The Course began on the first evening with a gentle reminder by David Watkin about essential mathematics in the so-called “Matrix Mixer”, and then ran for a further seven days with just an afternoon off for recuperation. Students were warned in advance that the Course would be intensive and it lived up to this promise.

Students came from a wide range – of places, fields of study and background – and had a similar range of experience of single crystal structure determination. The immense skill, effort and commitment by the group tutors in engendering co-operation and collaborative working is an essential part of the Course, and one which is very much appreciated by the students.

The Course covered all aspects of structure determination, including fundamental concepts of symmetry, diffraction, Fourier synthesis and direct methods and their application to solving and interpreting structures. The practical side also included a survey of crystal growth techniques, data collection, structure solution, refinement, interpretation and presentation. The Course finished with lectures and a practical session on databases given by staff of the Cambridge Crystallographic Data Centre.

The evenings were given over not only to the Matrix Mixer, but also to sessions on crystallisation, a crystallographic bar quiz, an expert panel and student presentations. The evening where the students were mandated to work together to produce group presentations was particularly entertaining and informative. For light relief one evening featured a Ceilidh marked by great enthusiasm: if occasionally the dances went chaotically wrong that was all part of the fun. The Course Dinner occupied the final evening, where those responsible

for making the Course a success – sponsors, organisers, lecturers, tutors, students and local staff – were thanked for their contributions.

The Course was clearly highly appreciated by the students, who provided valuable feedback which will inform the content and structure of the next Course in 2003. There are particular challenges in the wide range of previous experience of mathematics and crystallography. It is important to provide adequate support in these areas without detracting from the more advanced material, and this is one of several aspects of the Course which will be developed over the next two years. A book based on the Course, written by one past and three current lecturers and entitled "Crystal Structure Analysis – Principles and Practice" is due to be published by OUP in January 2002. The Ninth Course in 2003 will have five lecturers, with Simon Parsons changing role from tutor to lecturer.
The venue was again a highly successful element of the Course. The lecture facilities at Trevelyan College have been upgraded since the previous Course, and the close proximity to accommodation, catering and social amenities means that the different aspects of the Course could be easily integrated.

The number of scientists involved in crystallographic structure determination continues to expand: it is an essential technique but one where they must acquire expertise quickly and alongside other methods. The Intensive Course is a vital resource, offering the opportunity of concentrated study and learning support which is not available elsewhere. There is much use of crystallographic black boxes within computer programs, which invite the user to press a button or follow a script. In many cases this does work reasonably well but in difficult cases even the best black boxes may eventually say "Consult an experienced crystallographer". Only by understanding how these boxes work can difficult problems be solved, and developing this understanding is the prime objective of the Course.

**Sandy Blake**
**University of Nottingham**
**Course Director 2001**

## BCA Industrial Group Pharmaceutical Special Interest Group Meeting

**7th June 2001, GlaxoSmithKline**

Paul Higginson of Pfizer opened the meeting with the presentation "Automated Solutions to High Throughput Crystal Screening". Paul's presentation highlighted the cost that could occur in the pharmaceutical industry should the wrong solid form be taken into development. He highlighted the benefits of automated screening, not just to the researcher who is relieved of the monotony of repeated experiments, but also to the company who can reduce the risk of an unwanted form appearing late in development. Paul demonstrated the use of automated screening, and the instrumentation involved, with a successful case study. He concluded by emphasising that a high quantity of data needs high quality data management to produce usable knowledge.

"Prediction, Morphology and Mechanical Properties of Paracetamol" was presented by Sally Price of UCL. Sally's presentation demonstrated how difficult polymorph prediction can be, but also highlighted some of the ways that may make polymorph prediction more feasible in the future. She highlighted how the use of global minima in lattice energy would always be necessary, but pointed out the high number of minima that normally occur within a feasible range. She then went on to explain how you could eliminate unlikely structures based on their morphologies. For instance, crystal structures whose elastic constants mean they would be too compressible to be realistic are unlikely to form. In addition, crystal structures whose slowest growing face is too slow are also unlikely to form. Sally concluded by suggesting that involving nucleation kinetics may be the way forward in this area.

Our hostess Clare Anderton gave the final talk of the morning entitled "On-Line Monitoring of Solid-State Form during Crystallisations by Raman Spectroscopy". Clare explained what Raman Spectroscopy is and how it works. She then highlighted some of the advantages over methods such as XRPD, especially for online monitoring. These included: high throughput, real time investigation, multiplexing with more than one probe, portability

of the instrument and the sensitivity of Raman to solids forming in solution. Clare then took us through two case studies to show just how effective the method can be. The first case study showed how the crystalline form could be monitored from the mid-point to the end of a crystallisation procedure. The second case study showed how the form of a product varied during the manufacturing procedure, previously unseen by off-line measurements. Both case studies allowing quantification of forms to as little as 5-10% *in situ*.

The first presentation of the afternoon was by Dr. Arjen van Langevelde of Crystallics on "High-Throughput XRPD in Polymorph Discovery". Crystallics is a company which specialises in the investigation of solid form pharmaceutical materials, in particular polymorph identification, characterisation and production. Polymorph identification is achieved through an automated high-throughput crystallisation screen. Here concentration, solvent composition, temperature, cooling gradients and ripening time can be individually controlled to produce a wide range of crystallisation condition thus maximising the chances of discovering polymorphs. More than 1000 different crystallisation experiments can be undertaken at one time, yet each crystallisation takes place in 20ml wells, minimising the amount of material required. The resultant solids are screened for crystallinity by an automated X-Ray Powder Diffraction (XRPD) system, while proprietary software has been developed for the interpretation of the XRPD patterns to allow for fingerprint phase analysis through peak search and cross correlation. Once identified, Crystallics can characterise these polymorphs, off-line, by thermal analysis (DSC, TGA) and by crystal structure determination, typically using Single Crystal X-ray Diffraction. If necessary, a 'MultiMax' reactor can be used to optimise crystal growth conditions to produce suitable quality single crystals. If only small, poorly diffracting crystals are available the crystal structure can be determined from high-resolution XRPD data. *Ab initio* structure prediction is also offered as a means of structure determination. Finally, once polymorphs have been identified and characterised, the crystallisation conditions to optimise the production a specific polymorph on scale up can be precisely determined using the 'MultiMax' reactor in batches up to 50 ml. This system is able to automatically determine the meta-stable zone through solution turbidity measurements. (Further information can be found at http://www.crystallics.nl/)

Dr. Alastair Florence of the University of Strathclyde continued the session with his presentation entitled 'DASHtastic Adventures with the Bruker-D8'. Alastair related his recent success in using the DASH program to solve molecular crystal structures from Powder X-ray diffraction data collected on the Bruker-D8 diffractometer. The diffractometer in his laboratory is equipped with a primary monochromator to provide Cu $K\alpha_1$ radiation and a Position Sensitive Detector (PSD) which enables the accurate location of reflection positions along with good angular resolution and superior counting statistics. This specification produces data with a quality that maximises the chances of solving the structure. It is vital that the effects of preferred orientation on intensities are removed from the measured patterns and this is achieved by collecting data using a rotating capillary geometry. To reduce absorption effects borosilicate capillaries are used, while data is improved still further by using a 1mm slit which reduces the background at no cost to the measured intensities. Data analysis with DASH begins with unit cell and space group determination. The peak positions of the first 20 reflections are located using DASH and passed to an input file for a third party indexing program such as DICVOL-91. After indexing, a careful process of unit cell and space group verification takes places by visual comparison of predicted 'tick marks' *vs* observed peak positions. The next stage is to perform a Pawley fit of the raw data which seeks to model the pattern background, zero point, intensities, peak widths and also refine the unit cell parameters. Typically data is collected to 60° $2\theta$ but the high angle data is often omitted from the fit due to a high degree of peak overlap and poor signal-to-noise in this region. After data processing, the business of solving the structure can take place and this was described using Chlorpropamide as an example. Initially a trial structure was generated by placing the asymmetric unit at random within the unit cell. Comparison of the simulated XRD pattern with the

experimental one clearly demonstrated that the structure was incorrect. A Simulated Annealing algorithm was then used to move the molecules about the cell and vary torsion angles within the molecules to generate more trial structures and minimise the difference between simulated and experimental patterns. 300,000 structures were tested to find the one that gave an excellent fit across the whole pattern at which point the structure was considered to be solved. The process took just 5 minutes!

The third presentation of the afternoon was by Detlef Beckers of Phillips Analytical and was entitled "Temperature and Humidity Controlled X-Ray Diffraction Analysis on 4-Epi-Oxytetracycline". The ability to monitor structural changes of a material as a function of temperature and relative humidity is of great importance to pharmaceutical industry. An unexpected phase transition at high temperature and high humidity during storage or transport can turn a powerful medicine into a useless powder. Phillips Analytical have developed a stage for the X'Pert Pro Diffraction System where sample chamber temperature and humidity can be independently controlled. Humidity levels of 5% to 95% can be achieved between room temperature and 50°C. An example of the use of the system was described by reference to measurements on 4-epi-oxytetracycline (OTC). OTC is a basic compound for the production of various types of antibiotics, however it is unstable under ambient conditions and can transform to 4-epi-OTC. The level of the 4-epi-OTC impurity must be controlled to be less than 1% during production. HPLC is used to monitor the 4-epi-OTC level but the system must first be calibrated against standards of accurately known 4-epi-OTC content. In order to reproducibly generate these standard samples, the environmental conditions that govern the formation of 4-epi-OTC must be understood. Using the X'Pert Pro system, three crystalline phases were found to be associated with the formation of 4-epi-OTC and by recording PXRD patterns as a function of temperature (5°C steps) and RH (5 % steps) it was possible to construct a phase diagram relating all three of the phases. Using this information the conditions required to generate samples of precisely known 4-epi-OTC content could be determined.

Chris Weston of Bruker AXS concluded the session with his presentation on "X-Ray Rapid Screening System for Combinatorial Chemistry". Combinatorial chemistry refers to techniques to fabricate, test and store the resulting data for a material library containing tens, hundreds or even thousands different materials or compounds. Combinatorial investigations require rapid screening techniques to test and evaluate variations of composition, structure and property within a material library. X-ray diffraction is one of the most suitable screening techniques for solids since it is fast and non-destructive and abundant information can be revealed from the diffraction pattern. A two-dimensional X-ray diffraction system designed for rapid screening, D8 Discover GADDS for Combinatorial Screening, has been developed for this purpose. The system consists of a $\theta$-$\theta$ vertical goniometer on which are housed X-ray tube and optics, a two-dimensional X-ray detector, and an XYZ sample stage plus laser/video for sample alignment and monitoring. This geometry allows the combinatorial library of samples to be mounted with ease. The two-dimensional multiwire detector can collect a large area of a diffraction pattern with high speed, high sensitivity, low noise and derives intensities by integrating around the powder rings thus limiting the effects of preferred orientation. The laser/video system ensures that each sample is aligned accurately on the instrument center. Different areas of a sample can be probed by the system since the X-ray beam can be collimated from 1000 to 50 µm. Once all the data has been collected the GADDS software can be used to perform the user specified screen. This could be phase identification (qualitative and quantitative) or measurements such as degree of crystallinity, particle size, texture or stress. To achieve this a wide selection of screening parameters can be extracted from the patterns such as integrated intensity, maximum intensity, peak width, peak 2$\theta$ position, crystallinity and various stress components. The screening results can be displayed in colour coded map, 3D surface plot, or pass/fail map with user defined criteria.

**Brett Cooper, Merck Sharpe & Dohme and Neil Feeder, Pfizer**

# *Forthcoming BCA Meetings*

## Industrial Group: Autumn Meeting - Crystallography in Industry

**Thursday 1st November 2001, 10am- 4pm**
**Pilkington, Lathom, Lancashire**

*Welcome and Introduction*
Jack Brettle, Head of Science Support, Pilkington

*Crystallography and X-ray Reflectometry at Pilkington*
Mark Farnworth, Pilkington

*GADD Sir! 21st Century Tools of the Trade - Applications of XRPD in the Characterization of Pharmaceuticals*
Anne Kavanagh, AstraZeneca, Macclesfield

*XRD Studies on the Thermal Stability of Electrodeposited Nanocrystalline Nickel*
Matthias Abraham, University of Oxford.

*Microstructure and Performance of Materials.*
Keith Rogers, Cranfield University

*Presentation of an Industrial Group Award to Ian Langford*

*Award Lecture - Zinc Oxide*
Ian Langford, Honorary Senior Research Fellow, Birmingham University

*Applications of XRD in the Imaging Industry*
David Beveridge, ILFORD Imaging UK Ltd

*Glass Content of Ground Granulated Blast Furnace Slag*
Ian Slipper, University of Greenwich

*Standards for Line Profile Analysis*
Steve Norval, ICI plc.

**Optional visit to the Pilkington Exhibition Area at 9.30 or during lunch**
The Pilkington Exhibition area is well worth a visit. The 9:30 option will suit those people staying overnight in local hotels. The exhibition covers glass manufacture and the innovative use of glass in a diverse range of products. A registration form is included with this newsletter. For more information contact Judith Shackleton (e-mail: judith.shackleton@man.ac.uk)

## Chemical Crystallography Group: Autumn Meeting Mesomolecular crystallography

**Wednesday November 14 - 2001, 10.35am - 4.15pm,**
**Aston University**

The meeting will focus on methods and experiences of structure solution, refinement and results for large small molecules. Invited speakers include;

Dr Andrew Burrows (University of Bath)
'*Co-ordination and Hydrogen Bond Interplay in Supramolecular Network Formation*'

Professor Martin Schroder (University of Nottingham)
'*Construction of Framework Polymers: Catenates, Helicates and Porous Materials*'

Dr Jonathan Steed (King's College, London)
'*Crystal Frustration - and how to Avoid It*'

Professor Richard Winpenny (University of Manchester)
'*Studies of High Nuclearity 3d-Metal Cages with Unusual Magnetic Properties*'

A registration form is included with this newsletter. Full details including a registration form are also on the CCG pages of the BCA website. The local organiser is Carl Schwalbe (e-mail: c.h.schwalbe@aston.ac.uk). Parking on site is limited, and the use of public transport or car-sharing is encouraged. Participants who need car parking space should contact the local organizer directly.

Offers of short presentations (particularly by post-graduates and post-doctoral workers) at the meeting should be sent to the scientific session organiser,

**Professor Paul R. Raithby,**
Department of Chemistry, University of Bath, Bath BA2 7AY, Tel: (01225) 826444 Fax: (01225) 826231
E-mail: p.r.raithby@bath.ac.uk

## PCG Workshop Introduction to the Principles and Practice of Rietveld Refinement

**IMPORTANT - CHANGE OF DATE and VENUE**
Please note that this Workshop will now be held as a Satellite meeting to the BCA Spring Meeting 2002, at the University of Nottingham.

The workshop will take place immediately prior to the BCA meeting, on Sunday 24 and Monday 25 March, 2002. The Workshop will conclude at the start of the main BCA meeting at lunchtime on Monday 25 March. Full details are still being finalised and will appear on the Workshop WebPage: http://www.isis.rl.ac.uk/Crystallography/RietveldWorkshop.htm

With this meeting the PCG will begin a series of tutorial workshops on powder diffraction profile refinement methods. This technique, much used and a vital component of much of physical crystallography, is very powerful but if improperly used can lead to problems both in the refinement process itself and in the resulting structural models. The aim of these workshops is to provide a general introduction to the method and its applications. They are aimed both at those new to the technique, particularly research students and post-docs, and those who feel the need for a refresher.

The first of these workshops will be held on 24 & 25 March, 2002, at the University of Nottingham, as a Satellite meeting to the BCA 2002 Spring Meeting. It will introduce the basics of profile refinement using the Rietveld method. The workshop will include introductory lectures, demonstrations and hands-on examples. Topics will include:

- principles of Rietveld refinement, including minimisation;
- crystal structure refinement and what it achieves, including the use of constraints and restraints;
- data collection strategies, including angle- and energy-dispersive techniques with both X-ray and neutrons;
- basic refinement strategies - how to give yourself the best chance to get the right result;
- an introduction to some of the software suites available.

Speakers will include Bill David (ISIS/UCL), Jeremy Cockcroft (Birkbeck) and Kevin Knight (ISIS). Further details will be announced on the meeting Web site as they develop.

Contact the organiser Chick Wilson (C.C.Wilson@rl.ac.uk) for more details.

## BCA Physical Crystallography Group and the IoP Structural Condensed Matter Physics Group

**Autumn Meeting**
**Applications of high pressure in structural studies.**
**Hosted and supported by the Daresbury Laboratory.**
**Daresbury Laboratory,**
**5 December 2001**

This one-day meeting will cover the wide-ranging applications of high-pressure techniques in the study of crystal structure. The meeting will include scientific presentations, accounts of the latest technical advances in pressure techniques for both X-ray and neutron diffraction and also spectroscopy, and the opportunity to view some of the advanced high-pressure kit currently being made available in laboratories and central facilities. Among the topics covered will be: high-pressure single-crystal diffraction in the laboratory using point and area detectors; developments in high-pressure single-crystal and powder neutron diffraction; state-of-the-art high-pressure powder-diffraction at synchrotron sources; high-pressure EXAFS; and opportunities in high-pressure spectroscopy.

Speakers will include:

- Andy Jephcoat (Oxford) - Raman Studies of Bonding Under Pressure
- Dave Keen (ISIS) - High-Pressure Single-Crystal Neutron Diffraction
- Bill Marshall (ISIS) - Neutron Powder-Diffraction at High-Pressure
- Mohamed Mezouar (ESRF) - High-Pressure Powder-Diffraction Using Synchrotron Radiation
- Simon Parsons (Edinburgh) - Uses of Area Detectors in Single-Crystal Diffraction at High Pressure

Registration will be free and local accommodation will be available if required. The deadline for receipt of registrations will be 16 November, but please register earlier if you require accommodation.

For more information on the meeting contact the organiser: Dave Allan (The University of Edinburgh; D.R.Allan@ed.ac.uk) or the PCG Secretary Chick Wilson (C.C. Wilson@rl.ac.uk). Full details and an on-line registration form are available on the meeting website: http://www.isis.rl.ac.uk/crystallography/HighPressureMeeting.htm

# Other Meetings of Interest

If you have news of any meetings to add to list please send them to the BCA Web Master cockcroft@img.cryst.bbk.ac.uk or to the Editor, josephinejutson@beeb.net

**October 18 2001**
Frontiers in Crystal Structure Analysis, (Annual Meeting of the Swiss Society for Crystallography), Yverdon, Switzerland, 18th October 2001 [Details via IUCr website]

**October 22-26, 2001**
International Symposium on Crystal Chemistry of Coordination, Organic and Supramolecular Compounds. Moldova, 22-26 Oct 2001. [website http://phys.asm.md/ISCrChem]

**October 25-27, 2001**
Pittsburgh Diffraction Conference, Cincinnati, Ohio, USA, 25th - 27th October 2001[website http://www.che.uc.edu/Chemistry/PDC/Pdc.htm]

**March 4-7, 2002**
10th Annual Meeting of the German Society for Crystallography (Deutsche Gesellschaft fuer Kristallographie / DGK) Kiel, Germany [Details from IUCr website]

**March 23-28, 2002**
9th International Conference on the Crystallization of Biological Macromolecules, Jena, Germany, [website http://www.conventus.de/iccbm9/]

**March 25 - 28, 2002**
BCA Annual Meeting, Nottingham University (Full details in December issue)

**August 6 - 15, 2002**
XIX Congress of the International Union of Crystallography, Geneva, Switzerland [email: iucr@kenes.com]

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# BCA Corporate Membership

**The BCA values its close ties with commercial companies involved with crystallography. To enhance these contacts, the BCA is pleased to announce that they are now offering Corporate Membership.**

**Corporate Membership is available on an annual basis running from 1 January to 31 December and includes the following benefits:**

- Up to 10 free BCA memberships for your employees.
- A 10% discount on exhibition stands at the annual Spring Meeting.
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- Two free full registrations to the annual Spring Meeting.
- Ten complimentary copies of the quarterly BCA Newsletter.
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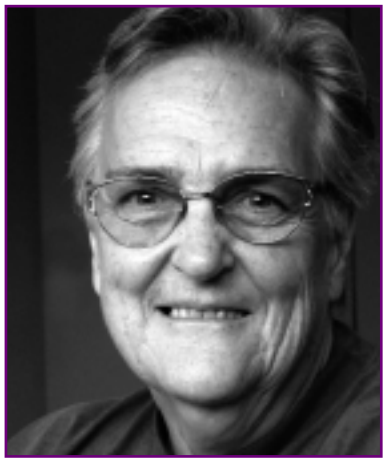
**e-mail** BCA@glasconf.demon.co.uk

# *The 2001 Walter Hälg Prize*

The recipient of the 2001 ENSA/Hälg Prize will be **Professor Jane Brown** of the Institut Laue Langevin, Grenoble, in recognition of her outstanding contributions to the science of neutron scattering over the last four decades. The prize is to be presented at a special session of the International Conference on Neutron Scattering, to be held in Munich between 9-13 September

Professor Brown has made a significant impact upon our understanding of the fundamental magnetic properties of materials through her contributions to both the development and exploitation of polarised neutron diffraction and advanced spherical neutron polarimetry techniques for the precise determination of complex magnetic structures and spin density distributions. She has played a key role in developing and establishing a computational framework, namely the extremely powerful and extensively used Cambridge Crystallography Subroutine Libraries (CCSL), to facilitate structure determination from crystalline diffraction. Professor Brown is also very well known to the European neutron scattering community for the expert guidance, support and training in single crystal and magnetic diffraction that she has tirelessly provided at the Institut Laue Langevin over the last thirty years.

Professor Brown is a graduate of Cambridge University, England.



Professor Jane Brown

She first became interested in the application of polarised neutrons in the early 1960s whilst spending two years at Brookhaven National Laboratory in the US. Returning to Cambridge as a senior assistant in research and subsequently assistant director of research in the Cavendish Laboratory and fellow and lecturer in Physics at Newnham College she forged close links with Harwell, where she established a programme in neutron diffraction. In 1972 she was appointed as Senior Scientist in charge of the Diffraction Group at the Institut Laue Langevin, Grenoble. She received a Gulbenkian Visiting Professor Appointment to work at the University of Coimbra, Portugal, in the early 1990s and has also been a Visiting Professor at Loughborough University in England for several years. Although formally retiring from the Institut Laue Langevin in 1995, she continues to run an extremely active research programme, whilst also remaining a very popular local contact for user experiments.

**Professor Bob Cywinski**
*Chairman of ENSA*
*University of Leeds, UK*

## PCG/SCMPG Bursaries

The PCG welcomes bursary applications from BCA or IoP members who are affiliated to the PCG or to the Structural Condensed Matter Physics Group of the IoP. These are intended mainly to help young scientists (students and post-docs) to attend meetings and conferences relevant to PCG/SCMPG areas of interest. Bursaries can be applied for at any time, through the PCG/SCMPG Secretary (C.C.Wilson@rl.ac.uk) and will be considered by the Group Committee. However, each year we expect to target selected meetings as highly relevant for the award of bursaries.

Applications for bursaries are expected to be received by the PCG Secretary **at least two weeks** prior to any early registration deadline - more details of deadlines will be publicised on the PCG Website. Recipients of bursaries are expected to write a brief report on the relevant meeting, and may be asked to report on a particular session.

**PCG Website:**
http://bca.cryst.bbk.ac.uk/bca/PCG/pcg.html

## CLRC e-Science Centre to run UK Grid Support Centre

The CLRC e-Science Centre has been invited by Tony Hey, the Director of the e-Science Core Programme, to set up and manage the Grid Support Centre for the UK on behalf of the programme. The CLRC e-Science Centre has also received funding from OST to develop a Grid infrastructure for its own experimental, computing and data facilities in support of its user community.
CLRC has invited Edinburgh and Manchester Universities to participate in the Support Centre to ensure that the Centre has access to the best experience currently available in the UK on Grid middleware and services.

When it is operational, the Centre will provide a range of services to researchers in the UK who wish to use the Grid in their work. The Centre will also be producing a report consolidating the experience of all 3 partners with the current generation of Grid tools.

For more information see website http://www.e-science.clrc.ac.uk

## Size Strain – III

***Analysis of microstructure and residual stress by diffraction methods***

December 2-5, 2001, TRENTO - ITALY
Information, on-line registration, on-line hotel reservation, instructions for authors, abstract submission, programme and updates are available only through the conference web-site:
*http://bragg.ing.unitn.it/sizestrain*

## Mineralogy Database

This mineral database website contains more than 5,000 web pages of mineral data. It includes selected mineral definitions and mineral pictures. There are 4,205 individual mineral species descriptions with links. Mineral data on individual species are linked to a mineral table by crystallography, X-Ray powder diffraction, chemical composition, physical and optical properties, Dana's new classification, Strunz classification, and alphabetical listings of mineral species. Links to other sources of mineral data available on the WWW are also included. Website address:
http://webmineral.com

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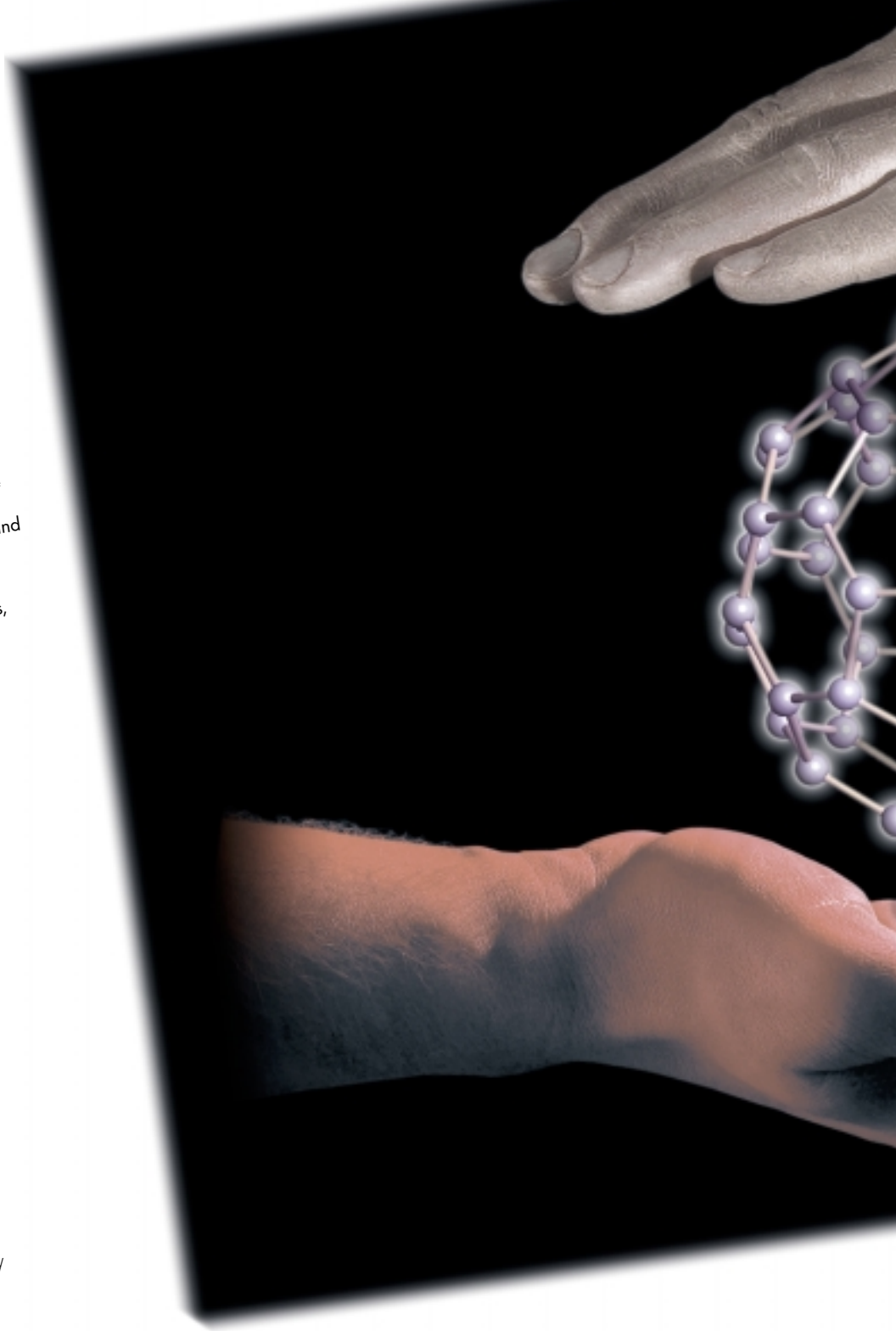

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- **Fast, accurate sample alignment and positioning** using sophisticated Laser/Video system
- **Precision mapping** using unique X, Y, Z Sample Stage
- **Automated operation** due to software batch script language
- **Applications** including compound analysis, compound ratios, amorphous and crystalline content, and crystallite size for catalysts, polymers, drugs, and other materials research
- **Structural information** by classic XRD techniques

# RAPID COMBINATORIAL SCREENING

## D8 DISCOVER with GADDS

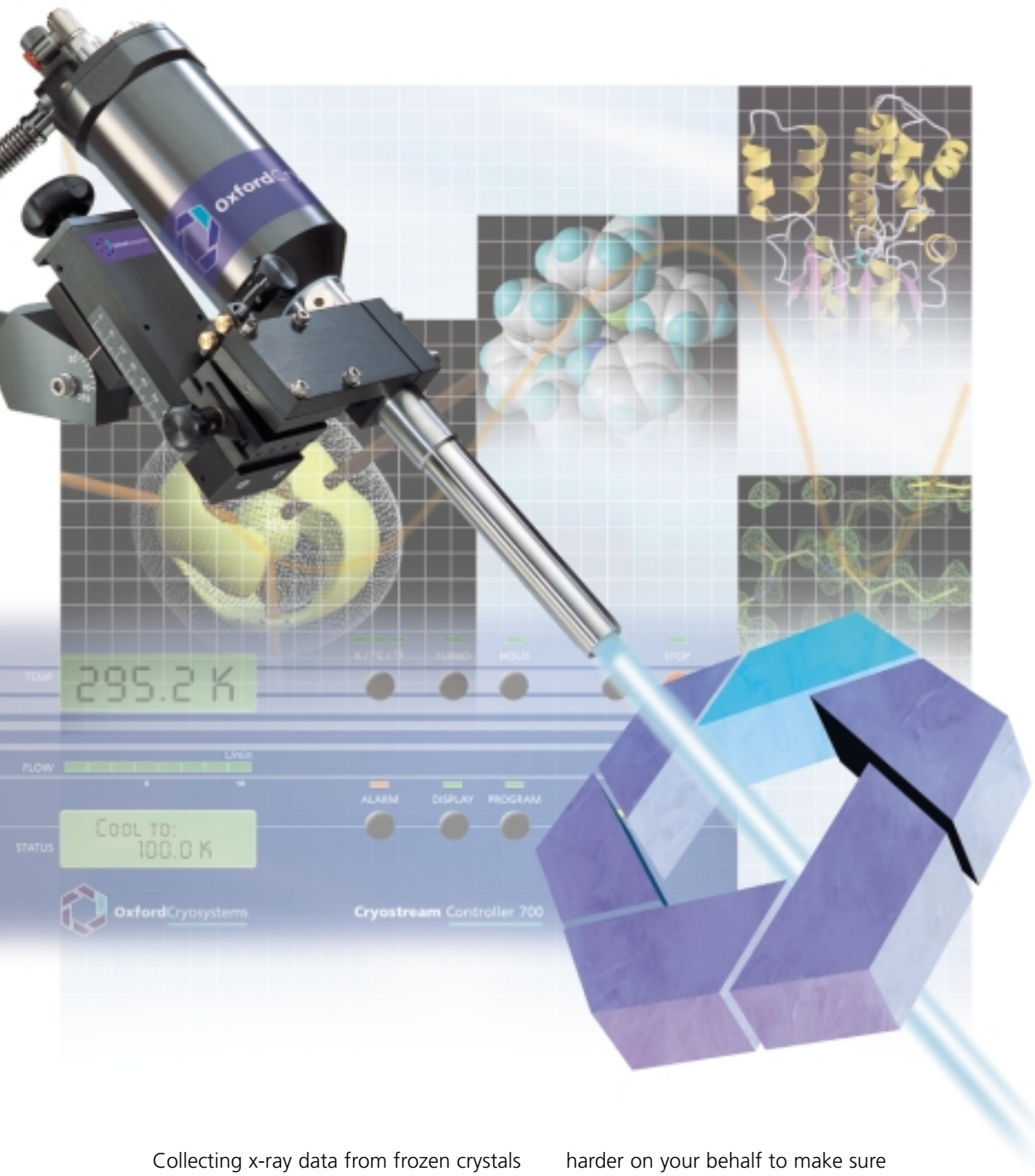
**Bruker AXS Ltd**
Tel: 01260 296900
Fax: 01260 296909
http://www.bruker-axs.co.uk

find out what's inside



BRUKER axs

# Cooler thinking...




Collecting x-ray data from frozen crystals can be a tricky business.

Mounting samples and freezing them needs careful attention to many variables all of which have to be just right to guarantee success.

When it comes to the Oxford Cryostream Cooler, we like to think just that little bit harder on your behalf to make sure everything goes to plan. With all of our new technologies and innovative ideas, we pride ourselves in our Cooler Thinking.

**OxfordCryosystems**

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