Crystallography News

British Crystallographic Association

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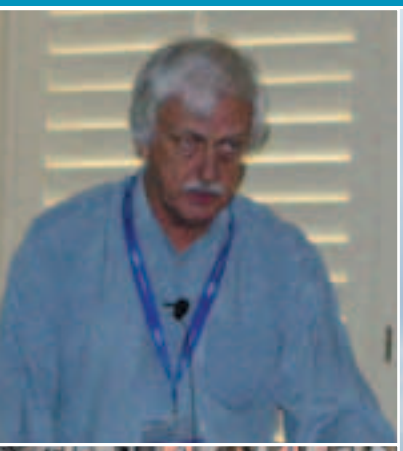

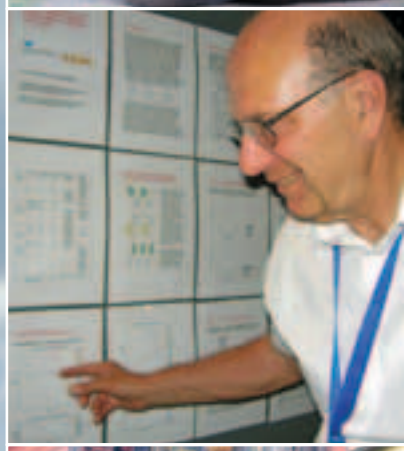

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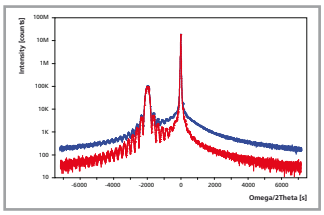

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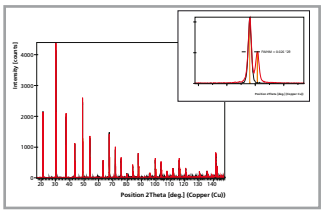

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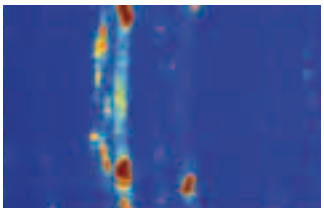

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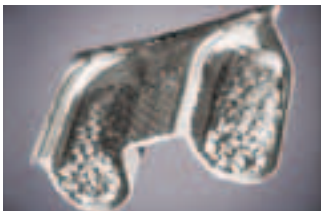

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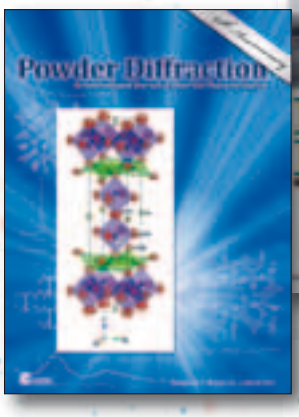

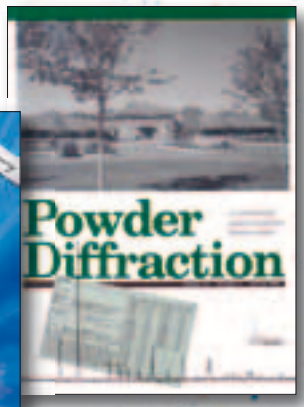

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Contents

This month's cover:

Scenes from the 2011 ACA meeting in New Orleans

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CRYSTALLOGRAPHY NEWS is published quarterly (March, June, September and December) by the British Crystallographic Association, and printed by William Anderson and Sons Ltd, Glasgow. Text should preferably be sent electronically as MSword documents (any version - .doc, .rtf or .txt files) or else on a PC disk. Diagrams and figures are most welcome, but please send them separately from text as .jpg, .gif, .tif, or .bmp files. 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 2011 issue are sent to the Editor to arrive before 25 October 2011.

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From the Editor

JAMBALAYA. To make this tasty New Orleans speciality you start by frying chicken, andouille sausage, celery, peppers and onions, then introduce more vegetables, tomatoes and seafood for further cooking, and finally add rice, stock and your favourite spices and give it a good simmer for up to an hour. The recent meeting of the American Crystallographic Association in New Orleans prepared its own very tasty version. Combine how-to-do-it sessions (workshops on symmetry-mode analysis and the PHENIX package, a session on how to get the best out of a synchrotron visit) with presentations of cutting-edge results (the Transactions Symposium on time-resolved crystallography and charge density in honour of **Philip Coppens**, lots of exciting structural biology) and discussions about how best to communicate our results (a session on publication procedures and the ever-popular "Would You Publish This?" event), spice it up with contributions from BCA members, and simmer the participants in record-breaking late May heat (97° F and very high humidity). The result left just about everybody satisfied. As I write this column in July, I am looking forward to the IUCr meeting in Madrid next month, where an alternative cooking technique using dry heat will be employed (I believe the Spanish word is asado).

Our cover and an inside page show some of the BCA members in action at the meeting along with scenes of New Orleans, where the Mississippi River was in full spate. If the Army Corps of Engineers hadn't opened the Morganza Spillway, diverting water from the Mississippi at the cost of flooding farmland, we'd have needed wellies. Please forgive me for including a picture of myself. Having learned about some of the additional features of my camera beyond simple point-and-shoot, I tried them out in New Orleans. The result of my attempt to outsmart the electronics was that most of my photos were under-exposed. The picture of me taken by Joan is one of the few that were correctly exposed. At least this lets me feel virtuous compared to the tabloid newspaper editors who have featured in recent news stories. I have only been adequately exposed to my own wife, and I have strictly limited the exposure of anybody else.

Following the large BCA Spring Meeting and the huge Congress of the IUCr, we can look forward to the BCA's much more intimate Autumn/Winter Group Meetings. Often these are the most exciting of all because everyone attending can contribute something to the topic of the meeting. I have to declare an interest here. My recent experimental research has involved comparing series of drug salts in which the counter-ions have regular increments to their functional groups. Such comparisons need structures for all members of the series, not just those that diffract well but also those with poor crystal quality, small crystal size and/or high Z'. This work has only been possible because of the power of the National Crystallography Service facilities and the Diamond synchrotron, facilitated by sympathetic help from the relevant staff. Therefore the forthcoming meeting of the Chemical Crystallography Group on "Dealing with Difficult Problems" will be just what I need. The other Groups will have stimulating meetings too. Check them out under "Meetings of interest".

At the time (late July) that I write this, there is still no resolution to the crisis over the U.S. debt limit. If the U.S. government runs out of money, nobody seems able to predict how it will affect science laboratories and scientists. Whatever the outcome of this situation, tough negotiations over the 2012 budget are a certainty. A report dated July 28 by Nature News describes the worrying implications: the worst-case scenario would be closure of one national laboratory or staff cuts at many. For the sake of colleagues whom I esteem highly and their very worthwhile research projects, I hope that the science budget will not be arbitrarily and formulaically reduced. Here in the UK we have many things to worry about, but we can be grateful that the government has expressed its appreciation of our major crystallographic facilities and appears to see the logic of continuing to get results from such major investments. While American crystallographers worry about possible man-made woes, Japanese crystallographers battle to recover from earthquake damage. The Photon Factory is making good progress. After careful safety inspection, test operation of the beamlines at the PF ring started on May 23rd. Some experienced users were asked to verify that their beamlines were working as well as before. Test operation of the PF-AR beamlines started on June 6th and has progressed similarly. Provided that sufficient electric power is available, it is hoped that user operation can restart after the pre-planned summer shutdown. Located further to the northeast, J-PARC suffered more damage. Impressive photographs have been made available on the Web (**http://j-parc.jp/picture/2011/07/J-PARC-Status-e110707.pdf**) that show its damaged facilities before and after repair has taken place. Water leaks were an early concern, a flood in the linac tunnel reaching a depth of 10 cm. Fortunately the leakage was stopped before components were damaged, but cooling systems still require major repairs. Shifting of the ground damaged electrical equipment and also caused misalignment of the 50 GeV ring beyond the adjustment limits of many electromagnet mounts. Despite valiant and successful efforts, much work remains to be done. We wish our colleagues a successful outcome.

Carl Schwalbe

BCA Council 2011

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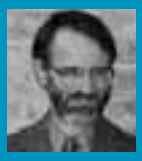

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(The dates in parentheses indicate the end of the term of office).

Full committee details on the BCA website **www.crystallography.org.uk**

Spring Meeting Registration and Subscriptions:
www.crystallography-meetings.org.uk

From the President

I start to write this letter with a heavy heart, having just learned of the untimely and sudden passing of Dr. Andres Goeta, a Senior Research Officer in the Chemistry Department of Durham University and a member of the BCA's Council since 2009. He will be very sadly missed, and we send our deepest sympathy to his wife, children, friends and colleagues. An obituary can be found later in this issue.

Andres had been due to be one of the five BCA delegates on two of the four evening IUCr Congress meetings at the XXII IUCr Congress and General Assembly in Madrid at the end of August, and he was keen to get a glimpse of the internal workings of the IUCr. The BCA will be represented at these meetings by combinations of the President, Vice-President, Secretary, immediate past Treasurer, and Council members **Alex Griffin**, **Arwen Pearson** and **Amber Thompson**. The Congress proceedings do indeed give an insight into how a multi-national organisation tries to carry out business and reach decisions: I was a BCA representative at the 2005 Florence IUCr Congress and the nature of the discussions and voting tactics enlightened me a little as to the challenges which must be faced by the United Nations Council as they attempt to optimise chances of peace and secure a safer world.

As those of you with an e-mail address known to the BCA will already know from my message of 14/7/11, the venue for the BCA 2012 Spring Meeting has had to be changed from Loughborough to Warwick, due to Loughborough University having been selected as the official preparation camp headquarters for Team GB and Japan. Loughborough can thus no longer accommodate our Spring Meeting. At such short notice, we are very fortunate that Northern Networking have been able to book us in to Warwick University for the same dates with similar arrangements as for SM2010. I am happy to say that although Warwick is a more expensive venue, Loughborough have said in writing that they will be covering the difference in cost. Therefore there should be no financial implications for the BCA. SM 2012 will be an excellent opportunity for us to have a 'dry run' of the venue before ECM28 in 2013. The planning for the meeting, led by Programme Committee Chair Dr. **Kirsten Christensen** from Diamond Light Source, is well underway, and further details can be found later in these pages.

We have now almost tracked down all the members to whom e-mails from the BCA bounce, but we are still seeking working addresses for the following members: **Ewald Schroder**, **Katie Sharp**, **Ravel Dokurno**, **Gareth Hall**, **Alan Hodgson** and **Mr. Ibarra.** If any of you have any information to offer, please let me know (**Elspeth.garman@bioch.ox.ac.uk**).

Also on the subject of membership, it appears that our 'Member get a Member' campaign (see my letter in the last issue and my e-mail of 28/6/11) is bearing some fruit, although at the moment the current President of the BCA is likely to be the front runner for the prize: free registration for the European Crystallography Meeting in Warwick in 2013. New members can now specify on their membership form who recruited them so that the recommending member can be entered into the competition. The winner will be announced at the 2012 AGM. Winning this would be a great embarrassment to the said President who dreamed up the scheme, so please would all members take this challenge seriously and recruit another member of their group or department. Membership of the BCA offers:

- Reduced registration for all BCA meetings,
- Four copies of *Crystallography News* per year to keep up to date with news and happenings in the field
- The opportunity to actively contribute to the development of crystallography,
- Networking opportunities
- The chance for students to apply to the Arnold Beevers Bursary Fund to obtain grants for meetings.
- The opportunity to interact and learn from scientists outside your own area of crystallography

We are also now offering an electronic version of *Crystallography News* to members as a pdf file instead of a paper copy. This will assist in keeping our costs down and is also better for our planet. If any member wishes to take up this offer please email the BCA Secretary **Georgina Rosair**: **secretary@crystallography.org.uk**

As some of you may already be aware, I have assembled most of the Archive material pertinent to the BCA in a filing cabinet housed in the subterranean depths of the New Biochemistry Department at Oxford. I was very pleased to be contacted recently by Dr. **Kersten Hall** from the Centre for History and Philosophy of Science, University of Leeds, who has just published a fascinating article entitled: *William Astbury and the biological significance of nucleic acids, 1938–1951*. In 'Studies in History and Philosophy of Science Part C: Studies in History and Philosophy of Biological and Biomedical Sciences.' Volume 42, Issue 2, June 2011, Pages 119-128, in which he examines the reasons why **William Astbury** was unsuccessful in solving the structure of DNA, even though he recorded an X-ray photo very like that taken by **Rosalind Franklin** two years before she obtained hers. Dr. Hall is gathering material for a further article on Astbury's life and work, and trawled the BCA archives last week, finding some very interesting information

about **Kathleen Yardley** who worked with William Astbury to compile the 230 space group tables which have since been hailed as something of a sacred text for crystallographers. He has by happenchance obtained access to much unpublished Astbury material through noticing that one of the librarians at his local library had the surname Astbury, and in chatting to him discovering that he was William Astbury's grandson. He then introduced him to other members of the Astbury family and thus he has found new historical sources.

Best wishes,

Elspeth

Cambridge Crystallographic Data Centre Now Accepts Structure Factors

www.ccdc.cam.ac.uk

THE Cambridge Crystallographic Data Centre (CCDC) is pleased to announce that structure factors are now being accepted with CIFs for deposition to the Cambridge Structural Database (CSD).

Colin Groom, Executive Director, commented that "The CCDC supports the International Union of Crystallography (IUCr) in highlighting the importance of retaining and validating experimental data. We strongly recommend that all journals publishing crystal structures should encourage their authors to provide these data in electronic CIF format."

"The deposition of structure factors to the CSD is extremely welcome and will greatly facilitate the work of reviewers" said **Sylvain Bernès**, co-editor Acta Crystallographica Section E. "Archived structure factors also promise to be a rich source of data for research related to statistical bias in x-ray structures and in detection of systematic errors in data collections".

CIFs and structure factors can now be deposited with the CCDC via our web-based deposition form at:

http://www.ccdc.cam.ac.uk/services/structure_deposit

Further information on publication standards can be found on the IUCr website:

http://www.iucr.org/index.html/leading-article/2011/2011-06-02

Dr. Gary Battle, Marketing and Communications Manager
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SYMMETRY TEASERS

THE much-appreciated BCA meeting at Keele University directed our attention to North Staffordshire and its best-known product, ceramics. The following images have been taken from the website of The Potteries Museum and Art Gallery, whose permission is gratefully acknowledged.

This museum has the finest collection of Staffordshire ceramics in the world and also shares ownership of the Staffordshire Hoard, the world's largest find of Anglo-Saxon gold. More images can be viewed at www.stokemuseums.org.uk. Each of the following plates and plaques shows some symmetry, but the symmetry is imperfect or only applies to part of the object due to the limitations of hand work or due to the artist's deliberate playfulness. Identify the symmetry of the whole or parts of the object and guess the ideal envisaged by the artist.

Thrown slipware dish, North Staffordshire, late 17 century.

Plaque by Charlotte Rhead, 1930's.

Thrown slipware dish, North Staffordshire, late 17th century.

Plate by Tom Arnold, 1971.

Plaque by Charlotte Rhead, c. 1935.

BCA Spring Meeting Reports

BCA Spring Meeting 2011

WE begin with accounts of the XRD and XRF sessions that have been written by our Industrial Group. We continue with summaries of sessions, presented in the same order as they appeared in the Abstract Book, that have been compiled from reports submitted by our bursary recipients, whose combination of enthusiasm and professionalism is gratefully acknowledged.

2011 BCA XRD Spring Meeting at Keele

IG Plenary

COMBINATIONAL STUDIES OF BIOMATERIALS

Nora H. de Leeuw, Department of Chemistry, University College, London

NORA began by saying mammalian bone and tooth enamel are organic/inorganic composites, comprising inorganic hydroxyapatite as the mineral phase and the collagen I protein as the organic phase. Due to its importance in natural bone tissue, hydroxyapatite is also used as a synthetic biomaterial, for example in hydroxyapatite/glass and hydroxyapatite/polymer composites. The talk presented an overview of the computational research carried out at UCL, to understand the structures and properties of a number of biomaterials.

D.F. Williams in 1987 defined a biomaterial as a material used in medical devices, intended to react with biological systems. For biocompatibility the chemistry of the implant and its leach-ability need to be understood. Nora mentioned that U.S.A. data for 2002 show that the overall medical device market is $77,000 million and that of this the biomaterials market is $9,000 million.

Natural bone contains a relatively simple protein phase (collagen). Nora showed high magnification images of the bone structure in which sheets of collagen fibres were clearly evident. Bone research presents many scientific challenges: 1) biomineralisation of organic/inorganic deposits, 2) design and integration of bioactive glass/ceramic implants, 3) the gap between experiment and computation, 4) the need for high accuracy but also large systems need to be studied. Hydroxyapatite, bioglasses and collagen for soft tissue replacement are some of the materials being studied.

Nora described in some detail her work with hydroxyapatite, $Ca_{10}(PO_4)_6(OH)_2$. Its natural crystal structure is hexagonal and its laboratory, synthetic, structure is monoclinic. The hydroxyl groups occupy different positions within the structure. OH ordering is evident. Type A defects occur when carbonate, CO_3, groups are in the OH channel. Type B defects are due to charge compensation arising from calcium vacancies and charge compensation by hydroxyl and cation (e.g. sodium) groups. Carbonates occur in natural apatite. Apatites in synthetic and high temperature apatites contain Type A or Type A and B defects whereas apatites in bone and enamel contain Type B defects.

Investigations of silica-hydroxyapatite interfaces have taken place. For hydroxylated surfaces the adhesion energy converges to a single value which is independent of the substrate surface or film rotation. It was found that the strongest apatite adhesion is to a de-protonated silica surface.

Nora went on to discuss Phosphate Based Bioactive Glasses (PBG's). These are the third generation of biomaterial whereby they promote tissue regeneration yet degrade after tissue repair. Calcium increases the rigidity of PBG structures by slowing down the dissolution.

The collagen phase of natural bone has also been investigated. Initial work concentrated on hydroxyapatite nucleation in an aqueous environment and on collagen bulk behaviour. Better mathematical models had to be developed to produce a 3-D representation of the collagen matrix. This reproduces the experimental structure and water content.

Mark Farnworth, Pilkington-NSG, Lancashire

IG1 and IG2 were joint sessions between the BACG (British Association of Crystal Growth) and two BCA groups: the Industrial Group and the Chemical Crystallography Group. The organisers were **Sven Schroder**, representing the BACG, **Louise Male** from the CCG and **Anne Kavanagh** from the IG.

IG1 was chaired by Dr. **Sven Schroder** of Manchester University and comprised three talks covering crystallization in the nuclear and photographic industries, and new tools to assess crystal structures from the CCDC.

Dr. **Karen Harvey** (Harman Technology) spoke about the controlled crystallization of silver halides for use in

photographic materials. She explained how the product performance of film and paper are determined by the crystal, size, size distribution and chemical composition of the silver halide particles. For photographic papers, sub-micron, monosized cubes of silver chlorobromide are required. For films, the particles are larger, tabular and consist of silver iodobromide, with a core-shell structure in which the core of the particle has a higher iodide content than the shell. The design and manufacture of these particles involves double-jetting of silver nitrate and sodium or potassium halide solutions into a gelatin solution, with precise control of the composition of the growth solution.

Dr **Sophie Janbon** (AstraZeneca) presented a joint contribution with the CCDC (Cambridge Crystallographic Data Centre). A collaboration between the CCDC and thirteen companies interested in the structure of small organic molecules has resulted in a new tool to study the frequency of hydrogen bonding motifs. This is relevant to the pharmaceutical industry because typical drug molecules contain several hydrogen bond donors or acceptors. The basis of the propensity tool is that the most energetically-favourable hydrogen bond interactions will be formed. The tool will help understand polymorphism in drug molecules by indicating when a particular hydrogen bond motif is common or unusual, and hence suggest whether a polymorph is metastable. Sophie illustrated her talk with pharmaceutical examples of Ritonavir, a treatment for HIV infection, and Bicalutamide, an anti-cancer drug.

Dr **Scott Owens** (National Nuclear Lab) gave intriguing insights into the challenges faced by crystallization studies in the nuclear industry, and the key part these studies play in, for instance, manufacture of fuel rods, scale formation and the recycling of fuel. Manufacture of fuel rods involves the formation of uranium oxide powders from the gas phase, and these powders must have the correct morphology (tabular) and particle size to ensure high quality fuel is produced. Formation of 'crud' in the reactor cooling circuit can cause blockages, Understanding crystallisation is also important in dealing with spent fuel rods, whether for reprocessing or for sending to waste. Reprocessing starts with dissolution of the spent fuel rods followed by mixing and concentrating several waste streams, without allowing crystallisation to occur, before calcining to form precursors to new fuel rods. Dr Owens also showed some amazing footage taken by a remotely operated camera from pipes in and around the reactor.

Dr. **Louise Male** of Birmingham University chaired IG2, which consisted of two presentations on protein crystal growth, and the YC Prize Lecture.

Dr. **Patrick Shaw Stewart** of Douglas Instruments described the benefits of seeded crystallization of protein crystals and offered many practical hints and tips for success, based on twenty years of experience. He explained that use of seeds increases the success rate because crystals could be obtained from within the metastable zone, without the need to go to the higher supersaturations required for spontaneous nucleation. Recommendations included using ground seed particles, suspending crushed seeds in reservoir solution, freezing seed stocks and avoiding the waste of seed by contact dispensing. The use of cross-seeding (with other proteins, ground glass or zeolites) can also be effective. The difficulties caused by salt formation and the possible polymorphism of the proteins were also discussed.

Dr. **Fabrice Gorrec** of the Laboratory of Molecular Biology, Cambridge, described an automated and extensive screening process for obtaining protein single crystals using a variety of 'LMB screens'. The screens aim to cover a wide range of conditions to maximize the chances of success, but are sufficiently straightforward that the non-expert can perform them. Recent developments include the use of statistical design of the screens to cover a wide range of conditions in as few wells as possible. Dr Gorrec finished his talk with some examples of the structures solved at the LMB in recent years.

The winner of the IG Prize for the best talk given at the YC meeting was **Oliver Zeldin**, of Oxford University for his talk 'High-throughput metallo-protein analysis by microPIXE'. Oliver explained the basis of micro-PIXE (a high energy proton beam excites characteristic X-ray emission form the sample) and how it can be used to determine the identity and stoichiometry of the bound metals. Currently the application of the technique is limited by the number of samples that can be run in a day and Oliver is developing methods to overcome this constraint using high throughput sample preparation via a technique of ink-jet printing the samples onto polypropylene. He has printed samples in arrays of 12x12 and demonstrated that no cross-talk between the drops is occurring during preparation or analysis, and is now working to finesse the technique.

Anne Kavanagh and Louise Male

XRF Spring Meeting Reports April 2011, Keele

Introduction

THE 29th Annual BCA Spring Meeting was held at the University of Keele from the 12th to the 14 April 2011. Delegates were drawn mainly from UK Universities, commercial and industrial institutions. As in previous meetings, the programme was packed with plenary lectures, workshops and themed, parallel, sessions organised by the Industrial Group, Chemical Crystallography Group, Biological Structures Group and the Physical Crystallography Group. For the fourth time at a BCA spring meeting, sessions were also provided for the XRF community - organised by the Industrial Group. The breadth and depth of the meeting was such that everyone had to be selective in the choice of presentations etc that they attended. Reports on the IG XRD content have been presented above. The XRF Sessions were officially opened by the XRF programme coordinator - Dave Taylor.

Sample Preparation Workshop

Laboratory Session. Frederic Davidts weighs a sample for fusion.

The workshop started in a laboratory a short walk from the main meeting venue and allowed live demonstrations of various sample preparation techniques. These started with the preparation of fused beads by **Frederic Davidts** and this theme was continued by **Rainer Schramm**. Both demonstrators were able to answer lots of delegate questions as they gave their live demonstrations. Rainer then moved on to a demonstration of the preparation of pressed pellets. All in all this was a very useful session with lots of delegate interaction.

Laboratory Session. Rainer Schramm describes fusion of samples.

Workshop Delegates. Speakers - Left to Right on front row: Ros Schwarz and Margaret West.

Fortified by lunch, the afternoon session continued the sample preparation theme as **Margaret West** considered the expanding range of techniques for the preparation of samples in the laboratory and in the field. Requirements for solids, powders, liquids & gels and environmental samples were discussed. Information on possible contamination from grinding media was presented as well as the consequences of particle size effects, surface finish of solids and liquid volume on the critical depth of analyte lines. Practical hints and tips were offered on the care of platinum ware and selection of sample support films. Delegates not wishing to discuss commercially sensitive information during the workshop found ample time for discussion during the evening exhibition buffet and breaks between sessions during the rest of the conference. After tea break, **Ros Schwarz** discussed some of the practicalities of setting up an XRF calibration. The features found in typical XRF spectra were outlined, for both WD - and ED – instruments, including tube lines and contaminants; and for ED-XRF sum and escape peaks. Line selection was linked to a consideration of excitation conditions and the instrumental parameters that are under the analyst's control, such as tube settings and beam filters; and for WD-XRF crystals and collimators. Line overlaps were illustrated and some of the ways to minimise the effects mentioned briefly. Some of the issues associated with WD-XRF, particularly second order diffraction from crystals and the role of pulse height discrimination in ameliorating the difficulties, were pointed out. As **Mark Ingham** was unfortunately unable to join the session, Ros also presented "Fun with background correction in WD-XRF". Several different strategies for background correction were introduced, using one or two background positions. A discussion of the determination of trace molybdenum at the single figure ppm level in a range of matrices showed that the Willis method of calculating a curved background from two positions gave the best results.

A delegate comment *"A long day, but full of practical ideas to take back to the lab".*

2. New Developments.

Speakers - Left to Right: Ros Schwarz (chair) Stefan Kneip and David Lane.

USING LOW COST CMOS SENSORS FOR X-RAY IMAGING AND SPECTROSCOPY

David Lane, Cranfield University

David began by saying commercially available X-ray sensors are available for scientific and medical applications. Whilst these can be large, sensitive and can provide energy dispersive information, they can also be expensive. Thus, the motivation for this study was to provide a low cost sensor with a USB interface for forensic applications in teaching, for scene of crime work and to screen suspect materials. The study examined the use of low cost CMOS sensors for X-ray imaging and spectroscopy. Examples of imaging using modified webcams were presented as well as those for a simple energy dispersive X-ray detector

based on an Omnivision OV7221 sensor, a device used in cellular telephones, PC multimedia and children's toys. X-ray sensitivity is enabled by replacing its front glass window with aluminium foil. X-rays are then detected as an increase in a pixel's dark current due to the generation of additional electron-hole pairs within its active region. Images of several minutes were achieved by recording the image as an AVI file, in which the energy information is retained through the brightness of each pixel in each individual frame. Frame stacking using image-processing software was used to simulate long exposure times. Black and white imaging was found to be the best so as to avoid the use of Bayer filters which are required for colour imaging.

BRIGHT SPATIALLY COHERENT SYNCHROTRON X-RAYS FROM A TABLE-TOP SOURCE

Stefan Kneip, Imperial College London

Stefan said that despite the demand, only a few dedicated synchrotron facilities exist worldwide, in part because of the size and cost of conventional (accelerator) technology. He described the use of a new generation of laserdriven plasma accelerators, which accelerate high charge electron beams to high energy in short distances to produce directional, spatially coherent, intrinsically ultrafast beams of hard X-rays. A focused high power laser beam is pulsed (30fs pulse width) into a plume of gas which becomes completely ionised. The source size is 1 to 2 µm. Hundreds of MeV electrons are generated which then produce tens of KeV X-rays with a spot size of 1 to 5µm.

The size of the synchrotron source reduces from tens of metres to centimetres, simultaneously accelerating and wiggling the electron beam. Stefan said that the resulting X-ray source is 1,000 times brighter than previously reported plasma wigglers and thus has the potential to facilitate a myriad of uses. We were shown how the tabletop device can be used to image biological specimens.

ENHANCING THE LIGHT ELEMENT PERFORMANCE OF EDXRF BY USING A NEW SILICON DRIFT DETECTOR WITH HIGH TRANSMISSION WINDOW

Colin Slater, Bruker

Colin described the S2 Ranger, 30 sample holder, benchtop EDXRF instrument. It has a palladium X-ray source with various primary beam filters and a silicon drift detector.The instrument has improved energy resolution and the stability of the detector over time is very good. To illustrate this, Colin said that a cement sample had been run every day for 77 days and there was no significant change in the data produced. Colin went on to say that the instrument is best in class for light element detection. The CEMENT quantification software package was described. It is pre-calibrated using a set of 15 reference materials.

Mark Farnworth, Pilkington-NSG

3. XRF/XRD Applications

Speakers - Left to Right: Nick Marsh, David Beveridge (chair) and Leah Cliff.

CHARACTERISATION OF THE PHOSPHATE SURFACTANT IN A LATEX USING XRF AND OTHER TECHNIQUES

David Beveridge, HARMAN technology Ltd.

The applications session began with a talk by **David Beveridge**, from Harman Technology, who described his work on the monitoring of the level of surfactant in latex by measuring its phosphorus content with XRF. He was able to use neat latex for the measurement, and calibrated using standards matched for matrix absorption coefficient made from disodium hydrogen phosphate, isopropanol and water. The results, expected to be 433 ppm, came lower at 300 ppm; so David turned to the venerable pH titration technique to investigate the puzzle. He found the two end points he expected from titration of the mono phosphate ester, which has two free -OH groups, but also that the titre for the second (at pH 8.8) was much higher than the first (at pH 4.7). This showed that the surfactant was actually a mixture of the mono and di phosphate ester bringing the problem to a satisfactory conclusion.

ENVIRONMENTAL APPLICATIONS FOR A NEW BENCHTOP XRD/XRF INSTRUMENT.

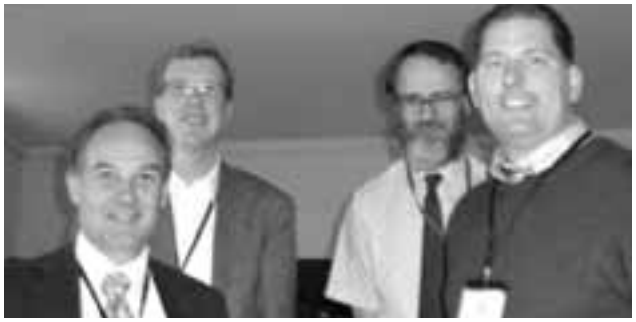
Leah Cliff, Southampton University

As an interesting contrast, our next speaker, **Leah Cliff** from the University of Southampton, told us about the CheMin instrument from InXitu which will be on the Mars Science Lab's "Curiosity" Rover to be launched in November this year. She mentioned Inxitu's three (terrestrial) combined XRD/XRF instruments, the Terra, Benchtop BTX and Duetto. The Benchtop BTX features transmission geometry and contains a 2D position and energy sensitive CCD detector to collect synchronous XRD and qualitative XRF data. The sample is presented in a vibration holder to improve particle statistics. She then discussed XRD data from 15 samples of hemipelagic sediments (a mixture of terrigenous and oceanic clays/sand/silt) taken from a Mediterranean seabed core spanning the last 1.1 million years. The mineral analysis was set up by reference to patterns from a laboratory XRD. The diffraction patterns obtained were comparable in resolution to those from a conventional instrument, but were limited to angles below 55° 2θ. The XRD results from the BTX were

compared to bulk XRD analysis and showed that the climatic modulation of the mineralogy is retained in BTX analysis, particularly in major mineral quantification.

Ros Schwarz, University of Sheffield

4. Applications (Two sessions)



Speakers - Left to Right: Phil Russell, Dirk Wissmann, David Beveridge (chair) and Christopher Shaffer.

APPLICATIONS OF HIGH POWER XRF IN GEOCHEMICAL AND ENVIRONMENTAL LABORATORIES

Christopher Shaffer, Thermo Fisher Scientific

Christopher began the second XRF applications session by highlighting the capabilities of WDXRF for routine trace elemental analysis in cases where ICP-MS, ICP-OES and other techniques are not suitable, often the case in environmental and geochemical laboratories where samples represent a wide elemental coverage, wide concentration range and varied sample matrices and sizes. Chris discussed continuous analytical advancements in WDXRF instrumentation making the method well suited to semi-quantitative analysis of low elemental concentrations (mg/kg) in liquids, rock/minerals and pastes. He introduced a new instrument, the Thermo ARL PERFORM'X Advanced WDXRF Spectrometer, capable of carrying out semi-quantitative, small spot analysis (5mm- 35mm) and mapping of non-homogeneous samples with minimal sample preparation. Chris also introduced the "UNIQUANT" analysis package for rapid analysis of totally unknown samples, requiring no preparation of standards. Multiple applications were presented, demonstrating the versatility of WDXRF alongside ICP methods, with applications ranging from oils and waste solvents, geological sediments, to cosmetics and food. Chris concluded by re-iterating that WDXRF cannot replace ICP methods but could instead work in collaboration with them, providing semi-quantitative identification and screening to facilitate full quantitative analyses using ICP methods.

NEW TEST METHODS FOR WDXF: TRACE ELEMENTS IN BURNER FUELS

Phil Russell, PANalytical

Phil discussed a landmark ruling in UK courts which redefined the description of petroleum products used for burner fuels, derived from waste lubricants, as a marketable product converted from lubricant waste oils. The ruling prompted new research into methods to develop and test burner fuels for conformance with Environment Agency quality controls. The performance of existing techniques (ICP-MS, ICP-OES, AAS) were compared alongside a high power, 3kW WDXRF, despite scepticism of WDXRF capabilities. Phil briefly discussed the preliminary round robin study, which demonstrated WDXRF suitability to the challenge over all other methods, using a 5ppm regulatory threshold and 0.5ppm detection limit for a range of elements. Subsequently, a full-blown round robin study (AAS, WDXRF & ICP-MS), involving 9 labs and 10 duplicate samples, was undertaken. WDXRF precision was close to the detection limit, revealing levels of performance not previously seen at such low levels (<5mg/kg) and demonstrating the stability in the WDXRF instrument and technique. WDXRF demonstrated better suitability than ICP-MS, which introduced errors by dilutions involved in sample preparation. In February 2011, methods for Burner fuels protocols also accepted test methods using WDXRF (except Hg) as a viable method for trace element analysis in burner fuels.

THE BALANCING ACT BETWEEN VERSATILITY AND ANALYTICAL PERFORMANCE WHEN USING ED XRF INSTRUMENTATION

Dirk Wissmann, SPECTRO Analytical Instruments GmbH

Dirk's talk discussed the historical uses of EDXRF spectrometers, predominantly in qualitative screening and quality-control applications. Dirk defined an ideal ED instrument, comparing and contrasting portable and lab EDXRF instruments to this 'perfect' instrument. He discussed universal calibrations and quantification of unknown samples by looking at Compton scattering, compared to mass absorption co-efficients for 1° excitation radiation, allowing calibration lines for silicate matrix correction and improved ED versatility. In the past 15-20 years, developments in detector technology (resolution, faster electronics) and optimised direct excitation have led to versatile spectrometers with greatly improved sensitivity for use in a broad range of applications; overcoming heavy-metal particulates in ship fuels for Al/Si analysis, trace metals in geological samples and elemental analysis (As, Cd, Sb) in PFO at 5ppm concentrations. Improved analytical performance in specific applications may be required more than great versatility. Dirk concluded that careful selection of components and hardware optimization can help fulfil typical requirements previously limited or impossible for ED XRF spectrometers. Consideration of Compton scattering in matrix corrections has allowed completely unknown samples to be analysed in minutes using EDXRF.

Leah Cliff, University of Southampton

Session Two

Speakers - Left to Right: David Bellis, David Beveridge (chair) Nick Marsh and Paul Vanden Brandon.

NOVEL APPLICATIONS OF XRF FOR MAPPING METALS IN INDUSTRY, HEALTH, AND THE ENVIRONMENT

David Bellis, currently unaffiliated

David Bellis spoke first describing collaborative work between the Wadsworth Center (New York State Dept. of Health) coordinated by Dr **Patrick Parsons** and XOS Inc, Albany, NY in developing new applications of XRF in health sciences. He described the use of a protype XRF mapping system to image lead and strontium distribution at the micro-scale in a bone slice, and how the instrumentation was subsequently developed for mapping of sectioned brain tissue. Other collaborations between NYSDOH and XOS in the development of instrumentation for measuring toxic metals in toys and jewellery, and toxic metals in blood in a clinical setting were also highlighted.

X-RAY FLUORESCENCE ANALYSIS OF ROCKS BY FUSION METHOD USING A BENCHTOP WD-XRF

Paul Vanden Brandon, Scientific and Medical for Rigaku

Paul Vanden Brandon presented work by Kansei et al. of Rigaku on the analysis of rocks via benchtop WD-XRF. The method targeted application in the mining/exploration industries, using small mobile instrumentation that can be installed in remote satellite locations. Fusion sample preparation was preferred over pressed pellets as it provided better calibration curves usign 10 certified reference materials from the Geological Society of Japan.

SUBSTITUTED TALK

Nick Marsh, University of Leicester.

Nick Marsh from the University of Leicester spoke about his experiences in preparing very small samples, often <100 mg material, for major and minor element analysis by XRF. The samples in question were residual sediment core samples previously analysed for C and O isotope ratio climate record, where diatom contamination by volcanic ash required correction by determining the completely elemental composition and performing mass balance correction of the measured ratio. Fusion preparation at high dilution levels was employed for sample preparation. Small quantities of certified reference materials were used to construct calibration curves, following additional grinding and calibration procedures.

David Bellis, curently unaffiliated

5. XRF Keynote - Margaret West

IT BEGAN WITH A HELPING HAND

Speaker - Left to Right: Margaret West and David Taylor (chair) chat after the conference dinner.

Margaret began her talk by saying that the novel techniques available today stem from **Wilhelm Conrad Röntgen**'s work in 1895. Since that time successive workers have developed ideas, built prototype devices and systems that have encouraged others to further explore the capabilities offered by the family of X-ray techniques. The word 'X-rays' has its origin in the German word 'X-strahlen' coined by the German scientist Röntgen. Röntgen was working with sealed glass tubes and his 'helping hand' was that of his wife, Anna, who placed it in front of an X-ray beam (!) which then produced an 'X-ray' of her hand on photographic film. In the UK between 1913 and 1914, Sir **William Henry Bragg** and Sir **William Laurence Bragg** were working with X-rays to look at crystal structures. They jointly received the Nobel Prize for Physics in 1915. During the early part of the 20th Century the X-ray Card Index system was being developed, originally under the JCPDS name and then subsequently under the jurisdiction of the International Centre for Diffraction Data (ICDD). **H.G.J Moseley** was a leading light in the field and he confirmed Bohr's theory of electron transitions.

During the 1960's automated wavelength dispersive X-ray Fluorescence (XRF) systems came on the scene followed in the 1970's by various mathematical models - **Willy de Jongh** (1973) and the Rasberry and Heinrich equations. During the 1980's microprocessor technology became more widespread and Energy Dispersive (ED) Systems became available. On the 2nd February 1990 there was an evening discourse at the Royal Institution when **Max Perutz** discussed 'blindfold automation' for problem solving!

In the modern era X-ray systems are available to suit all budgets, from sophisticated multi-technique systems to bench top to handheld. Total Reflection XRF is incredibly sensitive and has been a boon to the semi-conductor industry. Micro XRF is also available in which images of wafer contamination can be produced with 2D and 3D mapping of various elements easily carried out.

In tandem with recent developments in laboratory X-ray sources, synchrotron radiation has developed over the years. Combined techniques are seen more and more on beam lines. Among the many emerging techniques are

Time Resolved TXRF, X-ray Absorption Near Edge Structure (XANES) and X-ray Absorption Fine Structure (XAFS). 3D elemental mapping is becoming common place.

The Royal Society of Chemistry (RSC) support XRF through Atomic Spectrometry Updates (www.asureviews.org).

There have been staggering developments in computing power. There are now on-line data systems and fundamental parameter analysis is available to all. Detectors have improved tremendously. In the early days the detectors for EDXRF were bulky. They had an energy resolution in the range 170 to 200 eV. We now have Peltier cooled Silicon PIN or SDD detectors with an energy resolution of 130eV at ten times the count rate. Bench top instruments have 50W beam power and a low background signal. They are good for delicate samples. X-ray optics have also improved. We now have polycapillary systems and X-ray wave guides. The X-ray beams are smaller with higher special resolution.

Margaret concluded by saying that today we delight in sensitive systems, and that tempt researchers to explore new horizons. Where will it end?

Mark Farnworth, Pilkington-NSG

6. Novel Techniques and Applications

Speakers - Left to Right: Joanna Collingwood, Rainer Schramm, Adrian Wright and Dave Taylor (chair).

XRF ANALYSIS OF NEURODEGENERATIVE BRAIN TISSUE USING SYNCHROTRON RADIATION

Joanna Collingwood, Warwick University

Joanna set the scene of her talk by listing the range of degenerative neurological disorders with possible links to metal ions which included Alzheimer's, Parkinson's, CJD and MS. She then made some comparisons of staining with light microscopy, microprobe analysis and synchrotron microfocus XRF (at APS and Diamond). For microfocus mapping 10-12Kev is used for the transition metal elements of interest, including Mn, Fe, Cu, and Zn, and a multichannel detector is required to collect the full spectra with high sensitivity to the elements. This technique was able to locate the distribution of nanoscale particles and dilute elements across large areas of tissue, and allowed site-specific collection of XANES spectra to enable chemical and mineral characterization of the nanoparticles. Sample preparation methods include fresh-frozen sections, substrates of Permanox or quartz and Kapton coverslips. Joanna then went on to show the results from x-ray maps and outlined the advantages of the technique over conventional staining and the major challenges of the technique.

ISO 17025 ACCREDITATION FOR FUSED BEAD METHODS

Rainer Schramm, FLUXANA GmbH

Rainer followed on from his workshop demonstration of fused bead and pressed pellet preparation with a practical tour through the ISO 17025 standard for a fused calibration. His step by step approach from sample preparation, choice of calibration lines and backgrounds, calibration standards, measurement times and statistics, reproducibility and repeatability through to method validation with certified reference materials gave a logical understanding of the process to generate a good standardised analytical method. He went on to the explain the methods used to ensure that the calibration accuracy was maintained over time and left the audience with a good understanding of how to go about setting up a method to the standard required for accreditation.

CHALLENGES IN THE CHARACTERISATION OF AMORPHOUS METAL PHOSPHATES AND FLUOROPHOSPHATES

Adrian J Wright, Birmingham University

Adrian's talk focussed on the use of amorphous metal phosphates and fluorophosphates as bioceramic materials for bone replacement taking advantage of the properties of more stable amorphous materials providing a resorbable structure for new bone replacement and a stronger long term repair. The work centred on the formation of calcium, magnesium and strontium pyrophosphate as the target metals. XRPD, XRF and NMR were used to evaluate the reaction products as being amorphous (XRPD) with NMR confirming connectivity between phosphorus nuclei and WDXRF giving the elemental composition and proving the metal substitution. Comparison of fused beads to pressed pellets showed that fused beads gave more reliable results. Adrian then went on to discuss the poorly studied fluorophosphates, a common addition to toothpastes, and scope to generate new materials and the challenges of determining fluorine by XRF.

Dave Taylor, Session chair

7. XRF/PCG Cultural Heritage

The PCG/XRF session on Cultural Heritage at the 2011 BCA spring meeting was an absolute gem and if you missed it, you missed out! It nearly didn't happen as, less than 24 hours beforehand, we found out that two of the speakers and one of the chairs were unable to make it. But, after some hurried arm twisting two replacement speakers graciously agreed to step into the breach. And I have to say, they did us proud. The scene had already been set by a fantastic plenary lecture on cultural

heritage by **Gilberto Artioli** that morning and I was intrigued enough to forsake my usual biological structure group talks and stick around for the rest of the session.

CONSERVATION AND RESTORATION - SCIENCE BRINGS THE PAST INTO OUR FUTURE

Margaret West, West X-ray Solutions Ltd

The session opened with an interesting talk by one of our press-ganged speakers, **Margaret West** who, as well as giving the XRF plenary and running a workshop at the meeting, had kindly agreed to step in. She discussed her involvement in several conservation and restoration efforts around the UK. I was most intrigued by the idea of using XRF to identify components in paint in order to be able to formulate an exact match to a pot of paint that had been mixed back in the Victorian era.

THE ART OF INVESTIGATION: USING COMBINED DIAGNOSTIC TECHNIQUES FOR THE CONSERVATION OF CULTURAL HERITAGE

Liana Vella-Zarb, Heritage Malta

Margaret was followed by our second "surprise" speaker, **Liana Vella-Zarb** from Heritage Malta. Liana had managed to whip up a talk based on the poster she presented at the meeting that described the work of Heritage Malta in preserving the amazing array of historical sites on the island for future generations. I was fascinated by the story of her work on the ochre ceiling paintings in the Oracle room of the Hypogeum. This fascinating underground temple was rediscovered in the last century but the beautiful ochre paintings had appeared to fade over time. Liana explained how her group had used X-ray studies to determine that the ochre paintings were not in fact fading, but rather were being covered by a white deposit. This has enabled the group to establish a new approach to the care of the monument that should keep the paintings clear. It is on my list of places to visit!

A HOST OF GOLDEN ANGELS - COIN MAKING IN TUDOR TIMES EXPLORED BY XRF AND NEUTRON DIFFRACTION

Andy Smith, Daresbury Laboratory

The final talk in the session was from **Andy Smith** who presented his work on Tudor coin making. He was working on the astonishing array of gold coins that were found on the Mary Rose using X-ray and neutron studies to determine their composition. It was interesting to note that as Henry VIII fortunes waned, the gold coins become progressively less pure! However, Andy explained that there was a limit to how far the Tudor treasury could adulterate the coins. If the gold content was too low the coins became too hard to stamp with the face of the King. I very much enjoyed this session and am looking forward to sneaking into more non-bio sessions at future BCA meetings, I hope they will all be as interesting as this one.

Arwen Pearson, University of Leeds

BCA Spring Meeting 2011 Sessions

From Molecular to Supramolecular

THE "From Molecular to Supramolecular" session was chaired by Peter Byrne, and despite the lectures being in direct competition with George Sheldrick in a parallel session, managed to welcome a modest turn out. First up was Neil Champness (Nottingham) with his talk "Metal-Organic Frameworks: Applications in Hydrogen Storage and Photochemistry". Neil introduced these fascinating porous materials and their potential application to hydrogen storage and demonstrated the difficult issues in storing this gas in terms of the pore size, the interactions with Li and the balance between the two. He then moved on to discussing how metal-organic frameworks can be used as a matrix to hold photoactive species, in particular rhenium complexes which undergo isomerisation processes more reminiscent of behaviour in the gas phase – surprising as this is within a solid coordination polymer. The second lecture was given by **Andrew Cooper** (Liverpool), who provided an interesting paradigm shift in terms of porous materials, titled "Nanoporous organic cages - a modular alternative to nanoporous networks". These fascinating materials utilise non-connected organic cage-like molecules to induce permanent porosity through modulation of crystallisation solvent and composition of the cages themselves. One of the interesting properties of these materials was demonstrated by how switching solvents of crystallisation act as an on-off switch in porosity, thereby providing an active chemical trigger to change the properties of these materials in the solid state. The talk was finished by discussion of how these cage materials crystallise in the lowest energy arrangement, as from computer modelling calculations, and how these cages can be scaled up to an industrial level as these materials precipitate readily in phase pure form, thereby potentially providing a novel route for gas storage. The final talk of the session was given by **John Warren** (Liverpool), with the quirky title "Suck It and See". This was about how John was interested in understanding SO_2 uptake within 'Chinese-lantern' complexes, which led him to develop his own gas cell goniometer head to measure *in-situ* single crystal X-ray diffraction. Particular emphasis was placed upon the difficulties of using the gas cell, and the successes of using

this method with regards to the 'Chinese-lantern' complexes, and also metal-organic frameworks synthesised by the Rosseinsky group (Liverpool). It was particularly interesting to see how the presence of these gases can show changes in the structures of the materials under investigation, and how crystallography can be utilised to give information about structures which show such dynamic changes.

Jamie Gould, (University of York)

Twinning and Pseudosymmetry

Prof. **Eleanor Dodson** from the University of York chaired the session, which began with an excellent talk by one of the experts in the field, Prof. **George Sheldrick**. His talk about 'Handing small molecule and macromolecular non-merohedral twins' also explained clearly the complex problem often faced by crystallographers of merohedral and pseudomerohedral twinning. He gave examples to identify and solve the complicated issue by fine-tuning the parameters for indexing and integration strategies. This session was continued by Dr. **Roberto Steiner**. His case study of an oxygenase was the perfect choice to present a lecture discussing 'basic checks and practical advices when dealing with twinned data'. The advice covered technical and chemical options such as using additives that helped to resolve the problem by inducing different crystal packing and therefore the space group. Dr. Steiner further envisaged that twinning is very common, but experimental phasing is possible (MAD/SAD). However, the best way of solving the twinning problem is preventing the formation of the twinned crystals during the protein crystallization step rather than rescuing the data from the twinning troubles. The last talk of the session "Macromolecular order-disorder structures" was given by Dr. **Andrey Lebedev** from the University of York. He discussed several packing examples influenced by order-disorder structures from the literature and pointed out the possible ways where the twins been generated; these twinning cases can either be solved by detwinning or by relatively simple demodulation.

Helge Dorfmueller, (University of Dundee) and **Chao-Sheng Chen,** (University of York)

Protein Crystallization: dealing with low solubility protein:ligand complexes

This afternoon session, which imparted valuable experience of protein crystallization in the difficult case of low solubility protein:ligand complexes, was chaired by Dr. **Ray Owens** and kicked off with an interesting talk by Prof. **Zygmunt Derewenda** entitled "Engineering of proteins and complexes for crystallographic analysis" with the additional title "Macromolecular crystal engineering - from serendipity to rational design". He explained that protein crystallization is not a stochastic process and that there might be evolutionary pressure that some proteins will not crystallize. The macroscopic thermodynamic principles that govern crystallization can be understood in terms of microscopic structure, and further help to identify crystallizable proteins can be found in the protein sequence. Sequence related physical and chemical properties correlate statistically with crystallization success. Prof. Derewenda mediated to the audience that solvent accessibility has a great impact on that. However, from sequence alone surface residues cannot be predicted. One principle of improving protein solubility and thus obtaining a protein with much higher propensity is site directed mutagenesis. A case study showed that two mutants crystallized better than the native protein and Prof. Derewenda explained the rational design to engineer such mutants (Surface Entropy Reduction, SER strategy, see http://services.mbi.ucla.edu/SER/). The primary and secondary crystal contacts were explained and finally it was explained why the success rate of SER is unpredictable. A single patch SER approach can generate only one of the primary (cohesive) crystal contacts; however, multi-patch SER would be possible, but no guarantee for better prediction. Finally Prof. Derewenda emphasised that any modification has to be checked using for instance a fluorescence based assay to determine the melting temperature of the protein. Mutant proteins that reveal an increase in protein stability of several degrees are the most likely candidates to crystallize, but someone has to keep in mind the catalytic residues so as not to impair protein function. One question from the audience addressed the application of the SER method for membrane proteins. The application is limited to extracellular (soluble) domains of membrane proteins but not many examples are known to date.

Dr. **Elena Seiradake** presented the second talk of this session. She discussed her challenging research addressing the crystallization of a secreted mammalian glycoprotein complex. She very clearly explained in her talk the techniques to trim glycosylation enzymatically or by using cells with glycosylation-impaired expression. Further protocol optimisations led to crystals diffracting to higher resolution and gave insight into the extracellular domains of human Eph-ephrin receptor in complex with an ephrin ligand. Dr. Seiradake's efforts and methods to optimise a mammalian secreted glycoprotein were surely encouraging in particular to young crystallographers with regard to their own projects.

Helge Dorfmueller, (University of Dundee)

High Pressure and Energetic Materials

This session was chaired by **Christoph Salzmann** & **Matt Tucker**. The first presentation was given by **Colin Pulham** (Edinburgh), with the title "Modification of the structures of energetic materials using high pressures and co-crystallisation". This was a fun and inspiring presentation which highlighted the importance of energetic materials, using videos and a quick demonstration of how unstable they can be (inside the lecture theatre!). It was emphasised that how these energetic molecules are arranged in the solid state can alter the properties, making them more stable

for transportation, or potentially more volatile, depending upon the use. The introduction of secondary molecules, which can co-crystallise with the energetic molecules was also discussed, showing how the formation of more stable co-crystals can give rise to different properties of the materials in the solid state, in an almost analogous way to how different polymorphs of pharmaceuticals can change the therapeutic properties. Resistance to shock and impact were key in the energetic materials case, and hopefully the memory of the presentation will remain in our heads longer than the mark left by Colin's explosive on the table! Although **Peter Portius** (Sheffield) couldn't be with us to give the second talk of the session, as he had to give a talk at another meeting, we still got to hear about his research through his postdoc **Mark Davis**. Whereas the previous talk was concerned with controlling the stability of energetic compounds, this presentation was about the synthesis of greener compounds (those not releasing CO_2). Mark told us about how high nitrogen content was the key, with it all being hopefully converted to N_2. There was some tricky synthesis presented, but with some elegant crystal structures of hypervalent nitrogen-coordinating species. The structure-property relationship between the polyazido complexes investigated was also discussed in terms of energetics and stability.

Jamie Gould, (University of York)

Membrane Proteins

On the second day of the BCA spring meeting this year Dr. **Chris Tate** from the MRC Laboratory of Molecular Biology was invited to chair the session "Membrane proteins". Dr. **David Drew** from Imperial College London presented a talk about "Benchmarking membrane protein detergent stability for improving throughput of high-resolution X-ray structure". Acquiring a well-diffracting crystal is the bottleneck in studying membrane protein structure. Wise choice of the detergents was shown to be critical for stabilizing the membrane proteins; and it would be more likely to lead to high resolution structure with better stability. Dr. Drew also demonstrated a fluorescence-based unfolding assay which can be applied to benchmark the stability of target membrane protein. **Jennifer Miller**, a PhD student from Dr. Chris Tate's group then gave a talk about "Structural studies of a β_1-adrenoceptor: stabilizing the membrane protein by mutagenesis improves crystal quality". Jennifer had looked for several ways including introducing disulphide bonds, zinc bridges and salt bridges as well as leucine-scanning mutagenesis to improve the thermostability of turkey β_1-adrenoceptor and attempted to push the resolution beyond that of the published structure. Finally the engineered β_1-adrenoceptor can diffract to 2.1 Å demonstrating that improving the thermostability certainly helps the diffraction quality of the membrane protein crystals. The last talk of this session was contributed by Dr. **Stephen Muench** from the University of Leeds entitled "The mechanical and regulatory complexity of the vacuolar ATPase revealed." The techniques of electron microscopy (EM) and X-ray crystallography were both engaged in the studies he presented. The first 3D model of the tobacco hornworm V-ATPase had been solved by cryo-EM and allowed fitting the subunits previously solved by X-ray crystallography into the density map. Dr. Muench's talk demonstrated that the complement of EM and X-ray crystallography offers not only the overview of the large complex but also the significant details within the subunits.

Chao-Sheng Chen, (University of York)

Radiation Damage

In this session, chaired by Dr. **Colin Nave** from the Diamond Light Source, the president of the BCA, Prof. **Elspeth Garman**, gave a talk about "Radiation damage in MX: current challenges and new results". More often the radiation damage caused by the intense X-ray beams in the 3rd generation synchrotrons can destroy a sensitive crystal during data collection. Prof. Garman worked on the physical and chemical processes that produce the radiation damage and suggested that the protein active sites are more vulnerable to such damage. She also presented recent progress on investigating the use and radiation chemical mechanism of action of both nitrate and ascorbate as radical scavengers at 100K. The RADDOSE program can be applied to calculate the X-ray dose and was also illustrated in Prof. Garman's talk. Next, Dr. **Robin Owen** from the Diamond Light Source continued and presented a talk entitled "Running away from radiation damage: microfocus MX at room temperature". Cooling to 100K is a general way to reduce the radiation damage during data collection but still radiation damage is an issue due to the powerful X-ray beams as well as the fragile and sensitive nature of the macromolecular crystals. Recently, more challenging room temperature data collection was tested in the microfocus MX beamline I24, Diamond Light Source. Dr. Owen unveiled the progress optimizing room temperature data collection and further discussed the application of this technique to *in-situ* data collection and also the rapid diffraction screening of initial crystal hits in trays. The development of the high throughput diffraction screening can remove a hurdle in the crystallization pipeline.

Chao-Sheng Chen, (University of York)

CCDC / CCG Prize Lecture

The CCDC/CCG Prize Lecture was given by **Peter Wood** (CCDC), with the title "Teaching old dogs new tricks: using drug discovery techniques for crystal engineering", and was very enjoyable. Peter demonstrated how tools previously developed for specifically targeting prospective pharmaceuticals could be applied to various fields including porous materials, and the prediction of the structures of co-crystals from their prospective host-guest interactions. This work was fascinating not only due to its applications in such diverse fields, but also because of the potential impacts it could have in these fields. As a materials

chemist myself I found it entirely engrossing to discover how I could use such tools to predict potential separation properties within my materials by looking at the host-guest interactions of different molecules within a single framework. These ideas and the ability to recognise the potential of 'teaching his old dog these new tricks' made Peter a very worthy winner, and also made for a great lecture for the audience. Now if he only did something about that beard…

Jamie Gould, (University of York)

Dynamic Data; Dealing with Limited Data

With the evening of the conference dinner plentiful with drinks, new ideas, and talk of collaborative projects, it was sobering to enter the session titled "dynamic data; dealing with limited data", chaired by the CCDC/CCG Prize Lecture winner **Peter Wood**. The first presentation was given by **Iain Oswald** (Strathclyde), titled 'High pressure studies of chlorothiazide'. This was a good talk that reminded us of the difficulty of the presence of polymorphs within pharmaceuticals, and the importance of learning to control the way in which these compounds crystallise. In addition, it also reminded us of the challenges of trying to solve crystal structures from data collected using the high-pressure diamond-anvil cells. It was fascinating to see how Iain had managed to utilise the diffracted X-rays through the narrow window of his anvil cell to not only to solve a structure in a triclinic space group, but also to successfully identify a new polymorph, utilising both single crystal and powder diffraction methods. The second talk of the session was given by fellow Scot **Andrew Stewart** (Mainz) on solving structures using electron diffraction, with the title; 'Trials and tribulations of solving organic structures from 3D electron diffraction data'. Within this talk Andrew explained the method of electron diffraction and its inherent drawbacks, but also how the group in Mainz have overcome these problems. The result of this is a tool than can correctly solve structures and assign space groups when data collected by X-rays do not make sense compared to other structural techniques, such as electronic spectroscopic properties. Andrew finished his presentation by talking us through a case study of industrial importance, where the results from more standard crystallographic techniques could not explain the properties ascertained from the material under study. Only electron diffraction could give the correct structure. The final talk of the session was given by **Peter Smart** (Sheffield), a PhD student in the group of **Lee Brammer**. His presentation was titled 'Multi-step synthesis and reactions of metal-organic frameworks', and gave us a good insight to the difficulties in solving structures of these materials (tiny crystals!). Most of the talk was focused upon the synthesis of a two-dimensional material, linked by cobalt clusters. Interestingly there were coordinated solvent molecules pointing into the third dimension, which could be displaced by neutral ligands to change the framework from two- to three-dimensional. However, structural solution of these new formed materials is difficult as you can't rely on single crystal diffraction due to crystal cracking, meaning that only the Rietveld method can be used. This still gives reliable structure solution, and also a method for more modular synthesis, where the exfoliated layers could be combined with different functional molecules to yield new materials.

Jamie Gould, (University of York)

Hot Structures

One of the last sessions of this excellent meeting was chaired by Prof. **K. Ravi Acharya** and was covering 'Hot Structures'. These talks were exceptionally good, showing not only the insights into the structures obtained but also the long path towards the structure determination and interpretation.

Professor **E. Yvonne Jones** started the session with a great talk about the "Structural basis of semaphorin-plexin signalling". This ongoing research characterising the structural basis of cell-cell signalling events was dissected into the challenges of purification of this multimeric protein complex, liganded and unliganded complexes and their function and identified mechanism. The research conducted by her lab has led to the suggestion of a common mode of interaction that triggers all known semaphorin-plexin based signalling.

Prof. **Susan Lea** presented her research entitled "Structural studies into the control of type III secretion". Evidently the type III secretion system (T3SS) is a very important research area; in particular this system is under study in *Shigella flexneri*, a highly infectious bacterium. T3SS is composed of 25 proteins in *Shigella* that build a major machine into the inner and outer membrane and form an external needle. To date it is not understood how this secretion system is regulated and how effectors are regulated for secretion and injection into host cell cytoplasm. Also it remains unknown how many copies of each subunit are part of the ring structures, since it is difficult to determine the structure, even for EM studies. Prof. Lea's presentation focussed on a case study to answer the questions how many proteins form the ring structure of MixA, a membrane protein with a 700 aa cytoplasmic domain, 7-8 TMD. This protein is particularly interesting since deletion of domains and mutations in the probable functional domain stops secretion and therefore stops *Shigella* infections. Her case study can be summarised by her quote: 'You might be seeing your structure appearing in another journal; however don't give up, you might have the right crystal form that answers some unanswered question.' Prof. Lea's persistent interpretation of the structure has revealed some evidence for a mechanism that was validated *in vitro* and further on *in vivo* and led to a proposed molecular mechanism. However, many questions remain to be answered for the type III secretion system.

Helge Dorfmueller, (University of Dundee)

ACA AGM 2011

American Crystallographic Association Annual Meeting

AS usual, this was a very stimulating meeting. According to unofficial figures I obtained at the registration desk, there were 650 registrations, of which 128 were non-domestic. BCA members can be proud that 33 of these came from the UK, compared with only 11 from France. The Transactions Symposium always provides a focal point for an ACA meeting. This year's Transactions Symposium was entitled "Time Resolved and Charge Density" in honour of Philip Coppens, a most worthy recipient. No summary will be provided here since the Transactions eventually appear online with an enhanced article contributed by each speaker. Volumes from 2003 onwards can be freely downloaded at the following website: http://www.amercrystalassn.org/content/pages/main-transactions

The session on "Scholarly and Pragmatic Aspects of Crystallographic Publication Practices" was both authoritative, because of contributions from important editors of scientific journals, and thought-provoking, because speakers voiced their ideas frankly. Since it concerns every one of us who produces crystallographic results, whatever our sub-field, I am grateful for permission from ACA RefleXions to reproduce here the "official" account.

Carl Schwalbe

Scholarly and Pragmatic Aspects of Crystallographic Publication Practices

THIS important session fortuitously took place on the first day of the conference and enjoyed few empty seats. The presentations by an outstanding speaker line-up had a dual focus. On one hand, methodological aspects of preparing crystallographic results for publication in the scientific literature were covered in detail. The second thread addressed co-authorship policies, ethics issues, reviewer practices, and editorial experiences. It is fair to say that some crystallographers favor technical perfection whereas others use crystallography as a means to prove a chemical fact with structure quality, while important, relegated to secondary status. These two groups of people are united by a common desire to have the structures done "right." The placement of the threshold of technical acceptability on a scale whose endpoints are "Congratulations, there are no Alerts for your structure" and "Preliminary data, connectivity only" was a major focus of the session.

Mike Hoyland, actively involved in the data digitization at the IUCr, described the historical background of the CIF format -- the conceptual successor to the "standard crystallographic file structure" pioneered in 1984 -- which has evolved into an open access publication method in *Acta Cryst. Section E*. The use of checkCIF is necessary due to the sheer number of manuscript submissions to the IUCr journals; however, the alerts should be viewed as useful checkpoints rather than as a brick-wall-like barrier to publication. Whereas there are limitations to the validation algorithms, the latter offer tremendous help to authors, reviewers, and co-editors (think PLATON, checkCIF, publCIF). Future improvements to the existing structural tests and the implementation of new ones are expected on a continuous basis, and user feedback is encouraged.

Jerry Jasinski (Keene State College), who has published 33 papers to date this year, described the rigorous *Acta Cryst. C/E* manuscript preparation workflow followed by his undergraduate students. An important aspect of the process is to teach the students to avoid padding papers with "fluff" in order to expand their contents. If all authors submitting manuscripts to *Acta Cryst. C* and *E* followed the *Notes for Authors* as closely as is required of Jerry's students, the coeditors would have more time to spend on the structural science and chemical ramifications of the structures.

Richard Cooper (Oxford University) cast a strong vote in favor of the invaluable CheckCIF. Richard, curator of the crystallographic software package CRYSTALS, emphasized that CRYSTALS is a teaching tool and that it is imperative to train and educate crystallographers to evaluate the results objectively and to interpret structural peculiarities correctly. With a few reasonable assumptions one may expect over 95% of the structures with more than 1 level A/B alert to be correct. In the end, the data must support the structure, and checkCIF alerts should be understood and corrected or explained, rather than viewed as obstacles on the way to publishing a structure. As did other speakers, Richard gave a frank appraisal of some of the pros and

cons of CIF definitions and checkCIF tests, including the relative frequencies of valid and "false positive" alerts. On occasion checkCIF definitions may appear oversimplified or overcomplicated, in which cases Alert interpretation is not straightforward. An interesting question "Do different structures require different levels of validation?" certainly provided food for thought.

Bruce Noll (Bruker) confidently demonstrated the facile use of Bruker APEX2 software for preparation of crystallographic data for publication. Among other features, APEX2 can populate most CIF data fields with correct experimental data, run a quick and convenient data validation, generate a checkCIF report, and create an HTML-based structural report with an interactive JMOL molecular graphics window. In the case of well-behaved structures the CIF file created by APEX2 is suitable for deposition to the CSD and is essentially ready for submission to *Acta Cryst. E*.

It was a rare pleasure to hear **Judy Flippen-Anderson**, who, after light-heartedly requesting a cancellation of the session twice, delivered an evolutionary tale "From Independence to Service and Science to Technology" based on her experiences as a crystallographer, database specialist, and co-editor of *Acta Cryst. E*. In the (g)olden pre-PC days it was not unusual to see two papers per structure -- one crystallographic and one chemical. Later one chemistry paper combined both aspects, and now the structural details are frequently relegated to the supporting information. *Acta Cryst. E* papers have become glorified database entries and the quality of the papers in this journal has recently been slipping. Judy suggested that ways to improve the quality of papers could include requiring one author to have an entry in the IUCr World Directory of Crystallographers and restoring a short paper format with a mandatory Comment section, while maintaining the open access charge structure. Judy also showed interesting structural database deposition statistics -- the annual number of structures deposited with the PDB tracks the trend observed for the CSD with an approximate 20-year offset.

Robin Rogers (University of Alabama) gave an excellent presentation on the influence of culture on publication practices. For example, whereas copying masterpieces is a common undertaking in the world of art when students learn to draw, plagiarism is unacceptable in science. Unabashed copying of someone else's results is inexcusable; however, a different interpretation of the results may be welcomed. In chemical crystallography there may be a significant distinction between papers that report science and papers that report technique. If the technique is important its quality should approach the best attainable; if chemistry is the focal point then imperfections in the technique are forgivable. One should remain open to a diversity of ways of interpreting results in the appropriate context.

Arnie Rheingold (UC San Diego), who last attended an ACA meeting in 1993, spoke about finding standards for chemically important structures. When crystallography plays a supporting role there are caveats to interpretation of the results -- one must be objective and chemically knowledgeable. For example, symmetry may average six Cu–O distances in an octahedral Cu complex residing on a axis and "eliminate" the mandatory Jahn-Teller distortion.

From left: Mike Hoyland, Jerry Jasinski, Arnie Rheingold, Judy Flippen-Anderson, Bruce Noll, Larry Falvello, Ilia Guzei, Robin Rogers, Richard Cooper. Photo by Ilia Guzei.

Clearly, in this case one must interpret the disorder rather than report a novel and unique Cu complex without a Jahn-Teller effect. The most frequent source of complaints for a crystallography editor is the presence of thermally mobile and poorly modeled solvent molecules. If a solvent can be modeled and refined, then this is the avenue to follow; but after reasonable attempts fail to accomplish this task, PLATON/SQUEEZE is a respectable alternative. Importantly, the use of SQUEEZE, as well as the use of any constraints and restraints, should be thoroughly documented. Lastly, the co-authorship guidelines in the *Acta Cryst*. journals should follow the ACS standards -- all people who are responsible for the results should be listed as authors.

The final time slot was reserved for an open discussion, in which everyone present was invited to voice an opinion on the broad topic of publication practices. The discussion was spirited, with diverse questions and comments from the audience and the speakers. It was reiterated that authors should always give proper credit to all cited work even if the information is freely available from electronic sources. Individual structures from the CSD are preferably to be cited in place of a blanket CSD reference. **Peter Stephens** pointed out that structure validation gives a significant number of spurious warnings for powder diffraction analyses, including A-level alerts that are not applicable to powder studies – such as the absence of anisotropic U(ij) values. **Mike Hoyland** commented that more work is needed on structure validation for powder data. Another question addressed the alert generated by molecular fragments lying outside the basic unit cell. This stimulated a discussion about the optimum choice of asymmetric unit. In response to a comment by **Radu Custelcean**, **Ian Bruno** raised the question of whether it would be useful if each entry in the Cambridge Database had its own DOI. **Judy Flippen-Anderson** commented that entries in the Protein Data Bank have individual DOI.

The open discussion extended well beyond the scheduled closing time for the session. The speakers, one and all, had obviously touched on subjects of active current interest. And the topic of publication practices, which is presently undergoing important changes in both its technical and policy aspects, will undoubtedly continue to be actively debated among crystallographers.

This session was organized by **Ilia Guzei** (UW-Madison) and **Larry Falvello** (University of Zaragoza, Spain) and sponsored by the Small Molecule, Service, and General Interest SIGs, ACA, Agilent Technologies, Bruker, and the IUCr.

Larry Falvello and **Ilia Guzei** reprinted from ACA RefleXions

Fankuchen Award Lecture

ANOTHER high point of the meeting was this lecture given by the award winner and Honorary Life Member of the BCA, David Watkin. The Fankuchen Award honours contributions to crystallographic research by one who is known to be an effective teacher of crystallography. Studded with memorable quotes, this lecture addressed both research and teaching. Crystallography has gained an enviable reputation as the gold standard for structure determination. Historically, this was achieved mainly because practitioners knew what they were doing. In addition, the films that were necessary for data collection gave excellent insight into the reciprocal lattice. Nowadays there is a worry that computing has replaced thinking. Plainly, mistakes must be avoided, but how high should the quality standard be set? We do not want to be like Victorian crystallographers who precisely measured interfacial angles to seconds of arc, with very little lasting value. In effect we have a number of "Journals of Squeaky-Clean Structures". However, a "Journal of Rotten Structures" describing studies carefully done on difficult material would be interesting and could spur progress. Automatic structure validation based on physics is now well established in PLATON. MOGUL (from CCDC) provides some level of chemical validation, and could be used in the pre-publication stages of all analyses.

Considerations of cost require us to get the most structures from our equipment. About half the structures done at Oxford require only one working day from the start to publication-ready status. A common problem with the rest is disorder, which is the hardest thing for software to handle and a bottomless pit for human effort. We must set a limit on the maximum time spent on a structure analysis in view of its purpose: routine or really interesting. "We don't use a micrometer to measure the timber for a chicken house." Routine analyses can be done to an adequate standard. If a higher standard becomes necessary later, a new data collection can be performed to suit the new requirements. Simon Parsons suggests that one should spend no longer than about half a day on the treatment of solvent disorder. If the result is still not satisfactory and the disorder is only affecting solvent, SQUEEZE it out.

David posed a series of questions that we still need to address. Why and how does Nature choose space groups? Why do some structures have Z' > 1? Why are some structures modulated? How do molecules communicate with each other? This stimulating talk prompted a lively discussion. One point raised was that we seem to be seeing more structures with Z' > 1 nowadays, which may be attributable to the use of low temperature.

Carl Schwalb

Database Use and Crystal Structure Prediction

FROM disparate sessions I have picked out two lectures which illustrate the impressive progress being made in these fields. Thomas Womack described "Using Small Molecule Data for Ligands in Protein Structures". It is necessary to apply prior structural knowledge both to fit a novel ligand into the Fo-Fc map and to create reliable refinement restraints. The Cambridge Structural Database is an essential repository of such knowledge, but previous methodology that picked up bond distances and angles from a single structure could give a poor fit. More information is available: MOGUL supplies distribution output to match subgraphs of the molecule of interest. The GRADE software uses this output to produce restraints for the ligand that are compatible with the Engh and Huber EH99 restraints applicable to the protein. This information can be supplemented with quantum mechanical calculations of potential energy and its gradients. Transferring geometry, especially torsion angles, from small-molecule structures to protein-ligand complexes is not completely straightforward: protein binding may introduce strains not found in the small molecule alone, e.g. to make a salt bridge. Features such as stacked rings and 180° C-C torsion angles in aliphatic chains adopted for the sake of efficient packing in small-molecule crystals may not carry over to the protein-bound ligand.

In his talk about "Ab Initio Crystal Structure Prediction: Developments for Flexible Molecules and Applications to Porous Materials" **Graeme Day** demonstrated an iterative design process that started with the molecular structure, used crystal structure prediction (CSP) to generate the expected crystal structure and applied property prediction to calculate the materials properties. This information feeds into the design of an improved molecular structure. The CSP method using global lattice energy minimization is computationally demanding: with a single independent molecule one needs about 200,000 (quasi)random starting points, and about 1500 unique structures emerge from the energy minimization process. Nevertheless, we are now able to apply the methodology to cocrystals, a topic of particular relevance for the pharmaceutical industry. For instance, paracetamol forms a stiff crystal structure with poor compressibility but coformers can improve these properties. We need to answer the following questions: (1) will a cocrystal form, (2) what is its stoichiometry and (3) what is its structure? Studies on urea:acetic acid and caffeine:acetic acid have been successful. For flexible molecules one can use guidance from MOGUL to find appropriate torsions. Finally, predictable packing has been found for cage molecules.

Carl Schwalbe

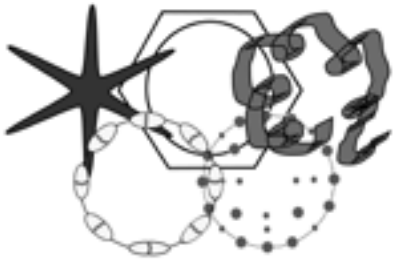
Top left: The Cathedral with the statue of President Andrew Jackson in front of it.
Bottom left: Close-up of Jackson.
Right: Ceiling of the Cathedral.

BCA Meeting 2012

BCA Annual Spring Meeting University of Warwick 16-19th April 2012 "Challenges in Crystallography"

AS announced previously by Elspeth Garman (BCA President) the University of Warwick will be the venue for the 2012 BCA Annual Spring Meeting. The meeting will follow the successful format used in recent years with the traditional Young Crystallographers' satellite meeting on Monday the 16th April, followed by the main meeting starting at 12:15pm on Tuesday 17th April with the final session finishing at 1:30pm Thursday 19th April. The overall theme of the meeting will be "Challenges in Crystallography".

Almost as important an event is the 2012 Olympic Games which is to be held here in the UK. We have therefore a sub-theme "Olympics of Crystallography", that is going to be reflected with a logo inspired from the five Olympics rings and the five groups within BCA.



Designed by Helen Maynard-Casely.

Like the Olympics, we will also have a medal ceremony at which the CCG and PCG prizes and the IG prize to a young Crystallographer will be awarded.

Dates, Links & Abstract Submission

All the latest information concerning the meeting, including the programme, registration and abstract submission, can be found at the meeting website: http://crystallography.org.uk/spring-meeting-2012.

Deadlines

This year we have a single common abstract deadline for oral and poster abstract submission including submission for the YC Satellite Meeting. **All submissions must be received by 16th January 2012**. If your oral abstract is not accepted, you will be invited to submit the abstract as a poster abstract instead. This deadline cannot be postponed due to the tight schedule surrounding printing the abstract book.

Currently Confirmed Sessions

TITLE	GROUP(S)	CHAIR(S)
Proteases in Disease	BSG	James Huntington
Drug Design	BSG	Rob van Montford
Hot Structures	BSG	Jon Cooper
Crystallography and Cancer	BSG	Jane Endicott
Structure to Function	BSG	Jonas Emsley
Protein Crystallisation: Magic versus Logic	BSG	Naomi Chayen
Dimensionality	PCG/CCG	Andrew Goodwin
Piecing Together the Puzzle – Multidimensional Approaches	CCC/YCG /PCG	Iain Oswald Anna Warren
Hydrogen Bonding: From Water to Supermolecules I	PCG/CCG	Christoph Salzmann
Hydrogen bonding: From Water to Supermolecules II	CCG/PCG	Peter Wood
Phase transitions I: Distortion Mode Analysis	PCG/CCG	Ivana Evans
Phase transitions II: Transformations in the Solid State	CCG/PCG /IG	Simon Coles
Software Workshop	IG/BSG	TBC
SAXS	IG/BSG	TBC
PAT – Online Processing	IG	TBC
Coatings	IG	TBC
Teaching Plenary: "Symmetry Modes: Nature's Favoured Description of Structural Distortions" (Branton Campbell)		
BCA Prize lecture, BSG Plenary & IG Plenary (Titles TBC)		

Programme Committee

Kirsten E. Christensen (2012 Programme Chair)
Ivana Evans (PCG)
Matt Tucker (PCG)
Helen Maynard-Casely (PCG)
Simon Coles (CCG)
Iain Oswald (CCG)
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Jonas Emsley (BSG)
Cheryl Doherty (IG)
Anna Warren (nèe Stevenson) (YCG)
Helen E. Mason (YCG)
Lee Brammer (2014 Programme Chair)
Elspeth Garman (BCA President)
Dave Allan (BCA Vice-President)

Kirsten E. Christensen, Programme Chair
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National Facilities News

IN the preceding issue of Crystallography News we noted the recent funding announcements for the continued support and development of both Diamond Light Source and the ISIS spallation neutron source. This is particularly welcome news as it offers security of operation for both facilities into the foreseeable future. For ISIS, the funding will allow the continued construction of beamlines for Target Station 2 and the upgrade and refurbishment of a number of instruments at Target Station 1. In this and following articles we will concentrate on the development of instruments at ISIS most closely associated with crystallography and structural science.

The developments at Diamond and ISIS are just the latest aspects of the evolution of both the Harwell and the Rutherford Appleton Laboratories which, as mentioned in the previous article, occupy the site of the former RAF Harwell airfield. Scientific activity on the site was initiated on the first of January 1946 when the Atomic Energy Research Establishment was formed to further the use of nuclear fission for both military purposes and for the generation of energy. An RAF airfield was chosen as the aircraft hangars would be ideal for housing the large nuclear piles, or reactors, and in addition an airfield would also be sufficiently remote, and have the necessary existing infrastructure, for a facility of this type. In 1954 the AERE was incorporated into the newly formed United Kingdom Atomic Energy Authority (UKAEA), which still owns much of the land on site. At its height, the Harwell Laboratory had five nuclear reactors and the first of these GLEEP (Graphite Low Energy Experimental Pile) ran for the first time on the 15th of August 1945 and was the first reactor to operate in Western Europe.

It operated for 43 years, a remarkably long life for a reactor, before it was eventually shut down in 1990 along with the remaining operational reactors on site. On the 14th of February 1957 the National Institute for Research in Nuclear Science (NIRNS) was formed to support research in nuclear science, which had developed beyond the scope and capabilities of a university laboratory. The first laboratory of the new institute, the Rutherford High Energy Laboratory (RHEL), was established on the site adjacent to the UKAEA Harwell Laboratory on a previously unused part of the original airfield. The first facility to be built at the new laboratory was the Nimrod proton synchrotron which, as we'll see later, is now the site of the ISIS spallation neutron source. In September 1979 the Rutherford Laboratory merged with the Appleton Laboratory (formerly the Radio and Space Research Station) to create the Rutherford Appleton Laboratory (RAL). In bringing the expertise of the two laboratories together on one site new space science programmes flourished and by 1981 the majority of the Appleton Laboratory had been transferred to the Harwell site from Ditton Park. The laboratory at Ditton Park was then closed after over 60 years of continuous research activity.

The use of neutrons for the study of materials at Harwell Laboratory dates back to the late 1950s, just after its formation, when the Dido and Pluto reactors went online. The reactors were heavy-water moderated and cooled, with a secondary light water cooling circuit leading to an array of cooling towers. They operated at 25.5 MW over a 24 day cycle with a four day shutdown period between each cycle for fuel changes and any necessary alteration to experimental equipment. The shutdowns were staggered by two weeks so that at least one reactor remained operating. The fuel rods consisted of tubes containing enriched uranium/aluminium alloy encased by aluminium. One of the main uses of the reactors was for the activation of materials placed within their cores. These materials were subsequently studied for the effects of radiation damage or their radio isotopes used as radioactive sources. The reactor vessels also contained apertures, or windows, to allow beams of neutrons to be used for diffraction and spectroscopy studies. In both reactors D_2O moderators placed close to the core "reflected" neutrons through the beam windows. On Dido there were four windows placed around the decagonally shaped reactor vessel. At face 5 there were two windows: one of these was used by the CURRAN powder diffractometer while the other was shared by the Mk VI 4-circle and 2-circle single-crystal diffractometers. The aperture at face 7 was shared by a 2-axis powder diffractometer, a triple-axis spectrometer and a beryllium filter spectrometer. To complete the set of instruments on the Dido reactor, a time-of-flight spectrometer was placed at the window of face 10. The Pluto reactor was built after Dido and had a slightly different design. Its reactor vessel had a simple square plan-form and there were also four windows available for diffractometers and spectrometers. At face 1 there was a small-angle scattering instrument while at face 2 one of the two available windows was shared by the MARX spectrometer and the SAD guide-tube diffractometer for small-angle diffraction. Finally, the aperture at face 4 was occupied by the PANDA high-resolution powder diffractometer.

Towards the end of the 1970s the Harwell 136 MeV linear accelerator, Helios, was made available for neutron diffraction and neutron scattering experiments. The accelerator fed pulses of electrons, at a frequency of 75 Hz, on to a water-cooled heavy-metal target which produced fast neutrons that were moderated in layers of hydrogenous material surrounding the target. The moderated neutrons could then be passed through any of the available 17 beam

tubes set in the structure of the concrete shielding to reach the spectrometers and diffractometers housed in two experimental block-houses at either side of the target station. At its peak of operation Helios supported 10 instruments which all exploited the time structure of the pulsed source to discriminate the energy, and hence the wavelength of the scattered neutrons, from neutron flight times over an accurately determined trajectory from the moderator, to the sample and then on to the detector.

The Helios facility demonstrated the potential of the time-of-flight technique and led on to the development of the much larger Spallation Neutron Source (SNS) facility following the winding-down of activities at Helios and both the Dido and Pluto reactors. At the heart of SNS is the site of the original 7 GeV Nimrod proton synchrotron, which was the last weak-focusing proton accelerator to be built. It operated between 1963 and 1977 and it had the principal remit of studying nuclear and high-energy phenomena. It was eventually closed down, along with all domestic high-energy physics experiments in the UK, in favour of an increased commitment to CERN and other large particle physics facilities overseas. With the decommissioning of Nimrod, the space around the accelerator mound and tunnels was used for the construction of SNS, which later became ISIS, and its first target station. As well as making use of equipment from Nimrod, the SNS also utilised components from the decommissioned Daresbury Laboratory NINA electron synchrotron, which was closed down in 1977 to make way for the Synchrotron Radiation Source. First neutrons at the SNS were produced at 7:16 pm on the evening of Sunday the 16th of December 1984 and, remarkably, within a few minutes the first test data were being collected and analysed. A first scheduled scientific programme of neutron scattering was initiated in April 1985 on the new facility – albeit at only ten percent of the machine's expected performance. In this initial period, six of the eventual eighteen neutron beamlines were provisionally instrumented for early commissioning. This first suite of instruments were: the Time Focused Crystal Analyser (TFXA), which was superseded by TOSCA in June 1998; the High Energy Transfer (HET) beamline; the High Resolution Inelastic Spectrometer (IRIS); the Liquids and Amorphous Diffractometer (LAD); and, of course, the crystallography instruments for single-crystal diffraction (SXD) and high-resolution powder-diffraction (HRPD). We will look at the SXD and HRPD instruments in some detail in the next issue. POLARIS, one of the original suite of instruments at ISIS, was initially envisaged as a development facility for neutron polarising filter devices on beamline S1. In 1989 it was converted to its more familiar role as a powder diffractometer and we will also catch up with the latest updates on this facility in the next issue.

The SNS facility was renamed ISIS soon after first beam. ISIS is not an acronym and it is actually the name of the principal goddess of ancient Egypt who was believed to have the power to restore life to the dead: she reanimated Osiris who had been killed and dismembered by his rival Set. Given that the original SNS had been assembled from the parts of preceding facilities, the name ISIS seems especially apt. Also, as the facility is situated close to the river Thames which changes its identity to the Isis as it flows through Oxford, the name ties the facility more closely to the region and one of its most prominent land-marks.

Next time we will look at some of the Target Station 1 instruments of most interest to the crystallographic community along with their most recent developments. I would like to thank **Mike Johnson** for allowing me to access some of his archive material which I'll be dipping into again for the next article. For those of you interested in the details of the some of the instruments available at Helios and the Dido and Pluto reactors at Harwell in the late 1970s, there is a booklet entitled "Neutron beam instruments at Harwell" edited by **A.H. Baston** and **D.H.C. Harris** of the Materials Physics Division of AERE, Harwell (which is available on the link **http://cdsweb.cern.ch/record/120067/files/CM-P00068619.pdf**).

The Dido (foreground) and Pluto (background) reactors viewed from the top of earth embankment behind ISIS. Both reactors had ceased operations by 1990.

One of the original Hangar buildings (Hangar 10) from the days of the RAF Harwell airfield. The roof of the building was raised in the winter of 1947-48, with the addition of the grey box-like structure, to accommodate the BEPO (British Experimental Pile) reactor.

The earth mound covering the proton synchrotron of the ISIS facility. The original NIMROD synchrotron was housed in the tunnels and containments covered by the earthworks. The heavily shielded access doors to the synchrotron tunnel are particularly impressive.

The building containing Target Station 1, and associated beamlines, of the ISIS facility.

The flags outside the ISIS reception and control room building. This was a good spot to enjoy the late evening summer sunshine while waiting for a taxi to take you to your (pre Ridgeway House) off-site accommodation.

Special Symposium Celebrating the 40th Anniversary of the PDB

IN October 2011, the Worldwide Protein Data Bank (wwPDB) will host a scientific symposium celebrating the 40th anniversary of the inception of the PDB, and the many scientific contributions it archives. <http://www.wwpdb.org/news/news_2011.html#14-February-2011>.

The program will showcase the scientific impact made by structural biology during the past 40 years with a distinguished panel of scientists who have been instrumental in the development of the PDB and structural biology.

The meeting will begin with an evening reception and plenary session on Friday, October 28th, and conclude with lunch on Sunday, October 30th, 2011. For details, please see the meeting website at **http://meetings.cshl.edu/meetings/pdb40.shtml**

Speakers

Cheryl Arrowsmith (University of Toronto, Canada)
David Baker (University of Washington)
Ad Bax (NIH/DHHS/NIDDK/LCP)
Axel Brunger (Stanford University/HHMI)
Stephen K. Burley (Eli Lilly & Co.)
Wah Chiu (Baylor College of Medicine)
Angela Gronenborn (University of Pittsburgh)
Richard Henderson (MRC Lab. of Molecular Biology, UK)
Wayne Hendrickson (Columbia University)
Mei Hong (Iowa State University)
So Iwata (Imperial College London, UK)
Louise Johnson (University of Oxford, UK)
T. Alwyn Jones (University of Uppsala, Sweden)
Brian Matthews (University of Oregon)
Jane Richardson (Duke University Medical Center)
Michael Rossmann (Purdue University)
Andrej Sali (University of California, San Francisco)
David Searls (Independent Consultant)
Susan Taylor (University of California, San Diego)
Janet Thornton (EMBL, Hinxton, UK)
Soichi Wakatsuki (IMMS-KEK, Japan)
Kurt Wüthrich (The Scripps Research Institute/ETH Zürich)

BCA Intensive Courses

The BCA Intensive Courses in X-ray Structure Analysis

IN September 2011 I will be retiring from the Chemical Crystallography, exactly 47 years after I took my first Weissenberg photographs under the tuition of Tom Hamor at Birmingham University. The BCA Intensive Course last Easter was also my last appearance on the stage there. At the final dinner, Judith Howard made some very complimentary comments about my contributions to the course, and the response from the students left me lost for words. I would like to take this opportunity to say that in reality I only played a small part in the Courses, and that their continuing success comes directly from a group of people enjoying working together with enthusiasm and commitment.

Before details get forgotten, it should be recorded that the Courses grew out of discussion involving many people, including Judith and **Chris Gilmore** (who I think was chairman of the CC Group at the time). My part was to take a proposal to Council to see if they would provide some financial assistance if we needed it, to which they agreed. EPSRC made a generous core contribution and 16 UK chemical companies provided additional funds. Judith and I organised the first Course, which was held in Aston University in January 1987 with **Carl Schwalbe** and **Phil Lowe** as local organisers. We had 16 lecturers and 8 tutors for the work sessions. The second Course was organised by a committee which also included **Bill Clegg** and **Bob Gould**. In succeeding Courses the numbers of lecturers was reduced in order to improve the continuity in the presentations, and as the work sessions became more important an increasing burden was put on to the tutors.

The Course rapidly won international recognition, so that places on it are still very much in demand both from UK students and ones from abroad. The success of the Course has been built in part on the dedication of the lecturers. These have been reduced to just half a dozen, but since they are all talking about their favourite topics to a captive audience, they seem to enjoy it. Possibly more important than the lecturers are the tutors. They have face to face contact with small groups if students and are expected to be able to help with problems from every part of the Course. It's very rewarding to see some of the tutors return later as lecturers, and many more move into established posts in the crystallographic community.

Crystallography is evolving, and the Course evolves to match it. There will be challenges ahead, and the sudden death of **Andreas Goeta** will leave a large gap. His amiable no-panic approach to every situation helped to make the Course a cheerful event. He will be sadly missed.

David Watkin

...Answer to March Puzzle Corner

TWO winners share the prize this time: (in alphabetical order) Bob Gould and Jim Trotter.

It is no surprise that this puzzle did not elicit many answers; difficult puzzles are rarely answered. Assuming that the camel doesn't mind coming back to a partly eaten banana, I would suggest that the answer is 153 333.. miles. With 300 bananas, it takes 5n one-way trips to move n miles. By going 20 miles, he would, after 5 half trips, be left with 200 bananas, 20 miles from the start. Now that there are only 200, it only costs 3n bananas to travel n miles, so he can travel 33 1/3 miles making 3 half trips and ending up with 100 bananas 53 1/3 miles from start. He then uses these 100 bananas to travel another 100 miles.

Bob Gould

My answer = 153 miles. Reasoning:
1. Camel Carl heads out for 100 miles, then realizes he has no way of getting back for the rest of the bananas.
2. Camel Jim thinks he is smarter, and after 50 miles decides to turn back for more bananas. However, he eats 50 bananas on the way back. After doing this again, he does 100 miles with the remaining bananas, and lands up at the same spot as Carl (but having eaten all the food, and covered 300 miles in total).
3. Camel Elspeth is more clever. She goes 1 mile, drops 98 bananas, uses the last banana to go back to the start; picks up another 100, goes one mile and drops 98 more, then back once more, and picks up the final 100 bananas. So she has gone 1 mile, eaten 5 bananas, and has 295 left. She repeats this process a total 100/5 = 20 times, so is now 20 miles from the start, with 200 bananas left. Then she starts moving one mile at a time again, but now requires only two trips using 3 bananas each time; hence she can go 100/3 = 33 miles, arriving at 53 miles, with 100 bananas left [she has had to leave one banana behind at mile 52, since there was no point in eating a banana to go back and get it, and then eat that banana too]. Finally she uses the remaining 100 bananas to go another 100 miles, total = 153 miles.
4. Camel Archimedes is a mathematician. He does what Elspeth did, but moves only an infinitesimal distance each time, eating an infinitesimal portion of a banana on each infinitesimal lap. Eating dx bananas moves him 1/5dx miles for the first 20 miles, 1/3dx miles for the next 33 1/3 miles, and then 100 miles on the final 100 bananas. Doing the integrations, he does 153 1/3 miles - but this is not very practical!

Jim Trotter

Obituaries

Dr Andrés Goeta 1965-2011

ANDRÉS GOETA came to the UK for the BCA teaching school in 1993 as a PhD student from La Plata, Argentina and was immediately popular and much admired. He stayed for the BCA meeting in Manchester that year and met many of our colleagues there for the first time. He later joined my group the following year, still during his PhD studies, in order to collect some low temperature data for his charge density studies as the machine at home was down for an extended period. He was funded by the British Council and he came with his young wife Marcela, for just a few months on that visit. He returned a year later in 1995 as a post doc in my group, from which he was appointed to the newly established post of X-ray Service manager for the department in 1998. Andrés trained as a physicist with Professor Graciela Punte in La Plata, but rapidly espoused chemistry. He was a very fine scientist and a real all rounder. He was a tremendous colleague and a very loyal friend to the group and the department. He has always been a great supervisor, mentor and teacher to our students, our post doctoral researchers and visitors from all over the world and nothing was too much to ask him to do.

He loved his work, his life in Durham, he was helpful to everyone in the department and beyond and Andrés truly inspired others by his attitude to his science and life in general. As well as his important managerial role for the department, Andrés was teaching the undergraduate and postgraduate students through lectures, workshops and laboratory classes. He had his own research group and PhD students but we all worked and shared our lab space and equipment together. Andrés took over the running of the long standing Durham BCA/CCG teaching schools recently and he has become a notable figure in the BCA community. He was a member of the BCA Council and would have been a UK delegate to the XXII Congress and General Assembly of the International Union of Crystallography, in Madrid later this month. Andrés had also been a co-editor of *Acta Crystallographica* for some years, but reviewed for many scientific journals. He was an excellent experimentalist and managed the X-ray service so well, at the same time as all of his research outputs. I cannot begin to tell you how much I and my group will miss him and the department too will feel his untimely passing in so many ways.

Andrés lived for his family and his work. He loved to do things *en famille* and with the children, hiking and camping all together in the Lake district, cycling locally or longer distance rides with his friends, swimming, walking his canine friend Max, an adorable chocolate brown Labrador, cooking for the family and friends and he had re-designed their kitchen recently to enable him to cook while entertaining, rather than to be shut off in the kitchen away from people. Summer BBQs were a favourite of his too. He loved to entertain and their house seemed always to be full of friends and their children.

Andrés has been tragically and so suddenly taken from us and the department has lost a real gem of a person and very clever scientist. Please add your own silent prayers and thoughts as you read this note and remember what Andrés meant to each one of you that knew him, in your own way. Our thoughts dwell principally with his family, his widow Marcela and their two children Annabella and Nico, his father Francisco and Marcela's mother Elsa.

Judith A K Howard, Durham Crystallography Group

The Passing of the Father of Neutron Diffraction in Europe

PROFESSOR G.E. BACON, the first neutron scatterer in Europe, passed away peacefully on the 18th March 2011 at the age of 93. He was born in Derby on the 5th December 1917, and educated at the 800-year old Derby school, and Emmanuel College, Cambridge.

George became Deputy Chief Scientific Officer at the AERE Harwell immediately after the war, and built the first neutron diffractometer in Europe. In a paper with **J. Thewlis** entitled simply "Neutron Diffraction" submitted to the Royal Society in 1948 he wrote "it is also shown that the powder method, which it is desirableto use when structure factors are to be determined, will be feasible if the number of pile neutrons which hit the monochromatizing crystal is greater than 105 per sec". In 1950 he described in The Journal of Scientific Instruments "Some Mechanical Features of a Double-Crystal Neutron Spectrometer".

Many papers on the application of neutron diffraction to both powders and single crystals followed, with evocative author lists such as "Bacon and Pease" and "Bacon and

Curry", such is the reputation of post-war English cuisine. Of particular note is his work with **R.S. Pease** in 1955 on the ferroelectric transition in Potassium Dihydrogen Phosphate (KDP) using single crystals polarised by an electric field. KDP was still being studied with neutrons decades later at Harwell and ILL by **Richard Nelmes**, **Werner Kuhs**, **Garry McIntyre**, **Christian Vettier** (at high pressure) and others including the humble author of these lines!

With **N.A. Curry** in 1956 George was the first to use neutrons for the study of hydrogen bonding in organic molecules such as alpha-Resorcinol, and with Curry and Wilson in 1964 the solid structure of crystals such as Benzene, which are normally liquids. **Stuart Wilson** was later one of the first British recruits to the ILL Diffraction group. George was also one of the first to use deuteration to study hydrogen bonding in crystals like hydrated Copper Sulphate. He was also interested in magnetism in materials such as K_2ReCl_6, later taken up by **Jane Brown** and **Bruce Forsyth**.

George's first book, "Neutron Diffraction" (1955) became the "bible" for early neutron scatterers, and ran to three editions and several languages. There followed "Applications of Neutron Diffraction in Chemistry" (1963), "X-ray and Neutron Diffraction" (1966), "Neutron Physics" (1969), "Neutron Scattering in Chemistry" (1977) and "The Architecture of Solids" (1981). His last book was as editor of "Fifty Years of Neutron Diffraction" (1987) which was also the title of the formal celebration in 1998 of his 80th birthday.

As a young man, George had worked throughout the war on the detection of the height of incoming bombers using the reflection of 1.5m radio waves. The idea was to use a vertical array of dipoles, producing a very narrow beam in the vertical plane due to interference effects, a little like x-ray diffraction in crystals. This effort resulted in a simple but effective "Chain Home" radar surveillance system of the entire coast in time for the Battle of Britain.

George became Professor of Physics at the University of Sheffield from 1963 until his "retirement" in 1981, but still visited Harwell, which he had helped establish as the first "user access" neutron source, for experiments. I met him 40 years ago when I was a young postdoc there with **Terry Willis**, the most unassuming and gentle man I have ever known. While I was struggling to understand the dynamics of structural transitions in KTN and KDP, I remarked to George that there were big changes in the powder patterns with temperature, if only we could extract that information. George smiled gently and remarked that some fellow in the Netherlands had been working on that recently. It turned out to be **Hugo Rietveld**.

Years later, well into his 80's, George was still working on D1B and D20 at ILL with **Pierre Convert**, on the texture of bones! A photograph shows him with **Alan Leadbetter** (UK Director) and Pierre Convert in 1998, but his last ILL experiment was in 2002. George was of the old school of gentlemen scientists, typically dressed in coat and tie in the laboratory. He believed in empirical methods, integrating Bragg peaks by drawing them on paper, cutting them out and weighing them, before Pierre Convert converted him to Macintosh computers when he was only 81. Young scientists today cannot imagine experimental physics before the arrival of the ubiquitous Personal Computer.

George was not a strong man at his remarkable age, so we joked about "Bacon's Bones". But he was onto something interesting as usual. He knew that the apatite crystals in bones were aligned for maximum strength, and that when a broken bone healed, it would reputedly never break again in the same place. So he wanted to know whether the crystals in the new bone were also aligned. Using neutron powder diffraction he showed that the new growth wasn't aligned; the extra strength was simply because the bone grew thicker at the break.

Then he asked about the apatite alignment in the various bones of animals, slaughtered at different ages after their bones had been broken and healed. Neutron diffraction was a great tool because it could penetrate thick materials. He found many examples of how nature had designed bones to be as strong and as light as possible. His most famous bone study was of prehistoric man. He showed that men who lived at the top of a cliff and had to climb up and down to the sea had stronger leg bones than those who lived by the shore and never needed to climb. These studies developed to fruition an approach initially suggested by his son Philip, a research biologist, who also helped with the early work.

George Bacon, the father of Neutron Diffraction in Europe, is survived by his son, and his daughter Jean, his wife Enid having died in 2003.

Alan Hewat, April 2011 (with **Sax Mason**, **Pierre Convert**, **Thomas Hansen** & **Philip Bacon**)

We thank Taylor and Francis for permission to republish this article, (2011) 'Obituary', Neutron News, 22: 2, 33 - 34.

GEORGE BACON was a much-appreciated colleague at Sheffield University. Despite his eminence, he remained a private and reserved person. Therefore there is no fund of colourful anecdotes about him - just an admirable record of diligence and scientific innovation.

After education at Emmanuel College, Cambridge (1936-1939), his proposed PhD at Cambridge was interrupted by the Second World War.

George joined the Telecommunications Research Establishment of the Air Ministry in 1939, continuing there until 1946. The first British experiments in radar had already been undertaken by **Robert Watson-Watt** in Feb 1935,

rapidly leading to construction of the first Chain Home stations during 1936-37. This first form of the technology was already defined by the time George joined the field. His early work involved helping to add a height dimension to the 'direction and ranging', a task which was completed rapidly enough to provide crucial altitude information to direct fighter pilots in the Battle of Britain a year later. Tests and refinements of the systems continued to be made throughout the war. George once remarked that he had asked over the radio for a Spitfire pilot to fly upside down in a test and found that the plane's radar signature reduced considerably. He also recalled that this may have been the first demonstration of stealth technology and noted than an American liaison officer was present at the time! He also wondered what the young pilots must have thought of the "boffins" who asked them to get up to these strange antics.

After the war George became Deputy Chief Scientific Officer at the Atomic Energy Research Establishment, Harwell. There he started his detailed work on the structure of graphite, not least because it was an important material at the time as a moderator in nuclear reactors. Because of the secrecy of that work, his eventual PhD was obtained as an external degree from London University. His thesis, entitled simply 'Some investigations of the structure of well-crystalised graphite by powder diffraction of X-rays and Neutrons', was examined by **J.D. Bernal** and **Kathleen Lonsdale** in 1952. However, neutron diffraction was soon to become his central theme.

The first edition of "Neutron Diffraction" appeared in 1955. The unique point about "Neutron Diffraction" was that George just sat down and wrote it and that it covered the *whole of the topic* of neutron scattering at that time. No one was ever able to do that again and that is what made "Neutron Diffraction" such a seminal work. With hindsight, it perhaps ought to have been called "Neutron Scattering", since it already covered inelastic scattering and small angle scattering. George expanded the later editions of "Neutron Diffraction" particularly the 3rd, but by then the topic was getting too big and a host of more specialist books by other authors had begun to appear.

The other point about "Neutron Diffraction" is its complete clarity and structure. The first part of the book describes neutron scattering processes and the fundamental advantages of using neutrons (*e.g.* low absorption, isotropic scattering, *etc*). There is then a summary table. This is followed by nine separate chapters which describe the different ways that neutrons could be exploited (*e.g.* study of light atoms in the presence of heavy ones; study of atoms with neighbouring Z; study of molecular structure; magnetic scattering, polarised neutron beams; inelastic neutron scattering *etc, etc*). Not only have these become traditional, but they have been used by generations of neutron scatterers ever since.

It was probably no accident that George should end up at Sheffield University. When he was exploiting the fundamental advantages of using neutrons described above, he often collaborated with scientists outside Harwell who could provide samples and expertise on the materials in question. In the 1950s and 1960s one of the hot topics (on both sides of the Atlantic) was the study of the magnetism in transition metals and transition metal alloys. The changes in magnetisation and magnetic structure type on alloying were of particular interest. George began a collaboration with **R (Bob) Street** at Sheffield, who subsequently emigrated and had a distinguished academic and research career in Australia and then with **John Crangle**, who incidentally followed George as Head of the Physics Department in the 1980s.

The period that George served at Sheffield, 1963-81, was actually quite a difficult one for Physics Departments throughout the country. Numbers of physics students were fairly static and Departments tried to make themselves more attractive by introducing various combined honours degrees. However, since most Physics Departments tried this option, it didn't do much except preserve the *status quo*. George served as Head of Department on three occasions 1963-65, 1969-72 and 1978-81 as well as one period as Dean of the Faculty of Pure Science 1969-71. He was much admired within the University administration for the professional and dignified way in which he always conducted himself, - which everyone who knew him will remember.

George's interest in the structure of bones was initiated when his son **Philip Bacon** did an undergraduate project on the form and strength of the bones of monkeys. A proper understanding of this topic involves the alignment of material in the dense 'cortical bone' of long-bone shafts compared with the open, fiberous (trabecular) structure of the joints. Philip guessed that the apatite crystals and collagen in the bone might be closely aligned and that apatite would be the more informative to measure. George must have known that there had been x-ray diffraction studies of apatite crystals in teeth in the past, including a letter published in *Nature* by his friend **J. Thewlis** in 1936. He quickly realised that the same ideas could be applied to bone, especially using neutrons for which the absorption would be low. Philip's supervisor **Rod Griffiths** provided some preliminary samples and George made the initial confirmatory X-ray measurements at Sheffield in the spring of 1975. He then borrowed a spare day of maintenance time on a Harwell diffractometer and made the initial neutron measurements later that autumn. George developed and refined the approach thereafter, using the more sophisticated 2-D and 3-D instruments at the I.L.L, Grenoble. These developments were greatly assisted by the enthusiasm and technical skills of **Pierre Convert**, who designed and commissioned the sample changers that were needed to take the drudgery out of this type of experiment, as well as the analysis of the subsequent data. He and George got along really well-- just two nice people who enjoyed working together.

George gave a colloquium on his work on bones towards the end of his time at Sheffield. He remarked that when a person starts a new and rather different project towards the end* of their career, it makes them realise that their scientific experience is literally *an integration* of all the things they have learned from all the different studies they have undertaken.
**except that he carried on for another 15 years!*

Neil Cowlam, **Philip Bacon** and **Carl Schwalbe**

Meetings of interest

FURTHER information may be obtained from the websites given. If you have news of any meetings to add to list please send them to the Editor, carlschwalbe@hotmail.com. Assistance from the IUCr website is gratefully acknowledged.

4-7 October 2011
Trends and Perspectives in Neutron Instrumentation: From Continuous to Spallation Sources, Tutzing, Germany
http://www.jcns.de/Workshop_2011

10-12 October 2011
GISAXS2011: Grazing incidence small angle X-ray scattering in thin film technology, Hamburg, Germany
http://indico.desy.de/conferenceDisplay.py?confId=4072

11-13 October 2011
Handheld XRF Workshop, International Centre for Diffraction Data, Newtown Square, PA, USA
http://www.icdd.com/education/handheld-xrf-workshop.htm

12-14 October 2011
ADD 2011. Workshop on Analysis of Diffraction Data in Real Space, Grenoble, France
http://www.ill.eu/news-events/events/add2011/

17-19 October 2011
Basic Rietveld Refinement & Indexing, International Centre for Diffraction Data, Newtown Square, PA, USA
http://www.icdd.com/education/rietveld-workshop.htm

20-21 October 2011
Advanced Rietveld Refinement & Indexing, International Centre for Diffraction Data , Newtown Square, PA, USA.
http://www.icdd.com/education/rietveld-workshop.htm

3 November 2011
Industrial Group Autumn Meeting, Harman Technology Ltd, Mobberley, Cheshire

3-4 November 2011
Physical Crystallography Group & Structural Condensed Matter Physics Winter Meeting, Applied Physical Crystallography, Coseners House, Abingdon
http://www.pcg-scmp.org/Meetings

8-11 November 2011
9th TOPAS Users´ Meeting with hands-on sessions, Bad Herrenalb, Germany
http://www.bruker-axs.com/topas_users_meeting_2011.html

16-19 November 2011
Multi-Pole Approach to Structural Biology: Recent advances of structural biology and bioinformatics, Warsaw, Poland
http://genesilico.pl/MultiPole/

29-30 November 2011
Perspectives in Terahertz Spectroscopy with Neutrons, Berlin, Germany
https://www.helmholtz-berlin.de/events/neat/index_de.html

16 December 2011
Biological Structures Group Meeting, Diamond

16-19 April 2012
British Crystallographic Association Spring Meeting, University of Warwick
http://www.crystallography.org.uk/

8-12 May 2012
ICCBM 14. 14th International Conference on the Crystallization of Biological Macromolecules, Huntsville, AL, USA.

31 May - 10 June 2012
Present and Future Methods for Biomolecular Crystallography, Erice, Italy
http://www.crystalerice.org/Erice2012/2012.htm

22-27 July 2012
15th International Conference on Experimental Mechanics, Faculty of Engineering, University of Porto, Porto, Portugal
http://paginas.fe.up.pt/clme/icem15/

9-13 September 2012
First European Mineralogical Conference (EMC2012), Frankfurt, Germany

25-29 August 2013
28th European Crystallographic Meeting, University of Warwick
http://www.crystallography.org.uk/

CCG Autumn Meeting 2011* "Dealing with Difficult Problems"

Wednesday 16th November 2011 Research Complex at Harwell

10:30 Registration (COFFEE/TEA)

11:10 Welcome & Introduction (Hazel Sparkes)

Session 1 (Chair: **Hazel Sparkes***)*

11:15 **Loes Kroon-Batenburg** (Utrecht)
"Unravelling and integration of diffraction data from crystals with twinning, disorder and distorted packing using EVAL"

12:00 **Zoltán Gál** (Agilent)
"Recent Developments in Indexing and Integrating Challenging Data"

12:30 LUNCH

Session 2 (Chair: **Paul Raithby***)*

14:00 **Richard Cooper** (Oxford)
"Dial-a-Standard Uncertainty"

14:30 **Horst Puschmann** (Durham)
"Olex2 and the smtbx"

15:00 COFFEE/TEA

Session 3 (Chair: **Iain Oswald***)*

15:30 **Helena Shepherd** (Toulouse)
"High Pressure Spin Crossover: How and Why"

16:00 **Claire Wilson** (Diamond / NCS)
"Don't Try This at Home Kids: Experiences from a National Facility"

16.30 CLOSE

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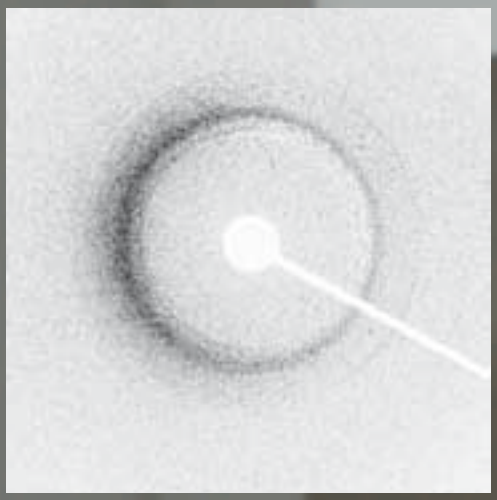

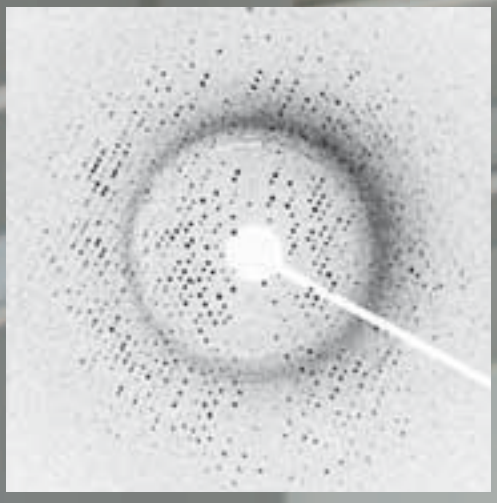

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